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THE EFFECTS OF FUEL ADDITIVES ON THE PRECOMBUSTION REACTIONS OF *n*-HEPTANE

By A. O. MELBY *

INTRODUCTION

KNOCK in a spark ignition engine is usually believed to be an explosive combustion of the end gas (the last portion of the fuel–air mixture to burn) which occurs when reactive species are stressed to critical conditions. These reactive species are formed by precombustion reactions that occur in the fuel–air mixture prior to and after the spark. In engines operating normally the study of precombustion reactions is complicated by the rapid movement of the flame front, which makes isolation of the unburned gases extremely difficult. This difficulty was overcome by Schmidt and Mühlner,[1] Damköhler and Eggersglüss,[2] and other investigators,[3, 4] who demonstrated that a non-firing internal combustion engine driven by an external power source was a very useful tool for the study of precombustion reactions. In such a " motored " engine, the behaviour of the entire fuel–air mixture simulates the unburned portion of the charge in a firing engine.

Recently, Pastell,[5] using a motored engine, defined the requisite physical conditions for the occurrence of the cool flame radiation and auto-ignition of *n*-pentane, *iso*-octane, and *n*-heptane, as well as for *n*-heptane containing varying amounts of tetraethyl lead. In addition, Pastell observed that a similarity exists between the resistances of hydrocarbons to auto-ignition in a motored engine and their resistances to knock in a firing engine. These findings indicated that the motored engine would be useful for a detailed investigation of the effects of antiknock compounds and other fuel additives on precombustion reactions. The initial phase of such a study has involved an investigation of the influence of tetraethyl lead, methyl aniline, di-*tert*-butyl peroxide, acetaldehyde, and diethyl disulphide on the cool flame and auto-ignition limits of *n*-heptane. This paper describes the effects which these additives produced.

PRECOMBUSTION REACTIONS AND THEIR DETECTION

A faint, pale blue luminescence or " cool flame " accompanies the vapour-phase oxidation of many hydrocarbons and other combustible organic compounds. The occurrence of cool flame radiation denotes that an intermediate stage in the oxidation has been reached. This has been shown by the spectroscopic studies of Emeleus [6] and Gaydon,[7] which have indicated that excited formaldehyde molecules are the source of luminosity. Walsh [8] has suggested that this excited formaldehyde may be produced by the reaction of two free radicals.

Observations of cool flames in bombs and tubes have usually been visual or have involved photographic procedures. These methods of detecting

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luminescence are impractical in a motored engine and, therefore, a photoelectric method was developed. This method is dependent upon the transfer of radiant energy from the reacting gases in the combustion chamber through a quartz window mounted in the cylinder head to a multiplier-type phototube. A 1P28-type phototube is employed as a detector, and its signal, after further amplification, is applied to the vertical input of a cathode-ray oscilloscope. Through this instrumentation the signal created by the faintly luminescent molecules is amplified 40 million times. This method and the detection equipment have been described more completely by Pastell.[5]

Experimental Methods

In this investigation the compression ratios required for the occurrence of cool flame radiation and auto-ignition were determined at various intake manifold air pressures using an ASTM supercharge method engine. The following procedure was employed :

With a fixed manifold air pressure, the compression ratio was increased from an initial low value, at which reaction did not occur, to that at which cool flame radiation was first evident on the cathode-ray oscilloscope. On further increasing the compression ratio an increase in the extent of reaction occurred, and ultimately, auto-ignition resulted. A similar procedure was followed at other manifold air pressures. At high charge densities (manifold air pressures of approximately 50 inches Hg abs and higher), the cool flame reactions passed so rapidly into auto-ignition that it was only possible to define one reaction limit. Cool flame and auto-ignition limit compression ratios which were less than 8·0 : 1 were reproducible within $\pm 0{\cdot}2$ of a compression ratio unit. Under more severe conditions (compression ratios greater than 10 : 1) the compression ratios for these phenomena were reproducible within $\pm 0{\cdot}5$ of a unit.

In order to evaluate the effects of the various additives on fired engine performance, octane numbers were determined. For these determinations, the engine was fired under conditions that were similar to those used for defining the cool flame and auto-ignition limits when the engine was motored. The same engine was used in all instances, and the octane numbers of the fuels were defined as being equal to the percentage by volume of *iso*-octane

Table I

Engine Operating Conditions

	Motoring for cool flame and auto-ignition limits	Firing for octane number determination
Fuel–air ratio	Stoichiometric	Stoichiometric
Compression ratio	4 : 1 to 22 : 1	Variable
Engine speed, r.p.m.	1800 ± 20	1800 ± 20
Spark advance, ° B.T.C.	Engine motored	30
Jacket temperature, ° F	211 ± 1	211 ± 1
Inlet air temperature, ° F	250 ± 5	250 ± 5
Oil temperature, ° F	160 ± 5	160 ± 5
Absolute manifold air pressure, inch Hg	20 to 85	30
Fuel system type	Manifold injection	Manifold injection

in an *iso*-octane–*n*-heptane blend which matched the knocking intensity of the test fuel. The octane numbers obtained differ from those that would have been determined by any of the ASTM knock rating procedures because the engine operating conditions were different.

Engine Operating Conditions. The conditions under which the ASTM supercharge method engine was operated are summarized in Table I.

Observations and Discussions

Fig 1 shows that tetraethyl lead increases the compression ratios required for the occurrence of both the cool flame radiation and the auto-ignition of *n*-heptane. However, the influence of tetraethyl lead on the lower cool flame limit is very small compared to its effect on the auto-ignition limit. Because of the relative magnitude of these effects, the

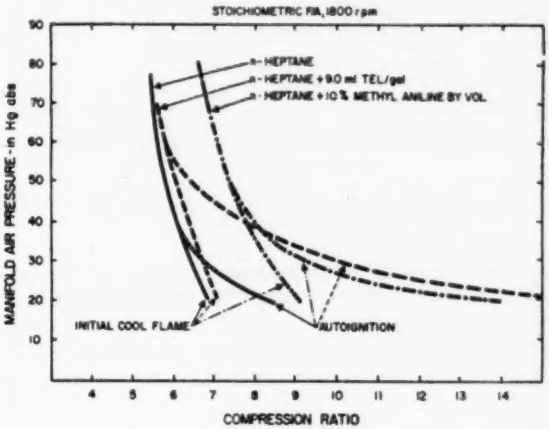

Fig 1

EFFECTS OF ANTIKNOCKS ON *N*-HEPTANE IGNITION LIMITS

range of physical conditions under which cool flames can exist is broadened. These data, therefore, indicate that the antiknock action of tetraethyl lead results from its ability to delay auto-ignition by prolonging the cool flame process. This prolongation, together with the small effect which tetraethyl lead produces on the lower cool flame limit, suggests that the decisive chemical reactions through which tetraethyl lead functions to suppress auto-ignition, and hence knock, occur during the cool flame process.

Methyl Aniline. It is apparent from Fig 1 that the antiknock action of methyl aniline is distinctly different from that of tetraethyl lead. Methyl aniline has a pronounced effect on the chemical reactions which lead to the formation of the initial cool flame. This effect is somewhat more apparent in Fig 2, where the compression ratios requisite for the occurrence of cool flame radiation of *n*-heptane in the presence of methyl aniline and tetraethyl lead under various manifold air pressures are plotted against the corresponding compression ratios which are necessary for the initial cool flame radiation of pure *n*-heptane.

Additional information concerning the mode of action of methyl aniline is shown in Fig 3. These data indicate that 10 per cent of methyl aniline by volume is more effective than a tetraethyl lead concentration of 9 ml

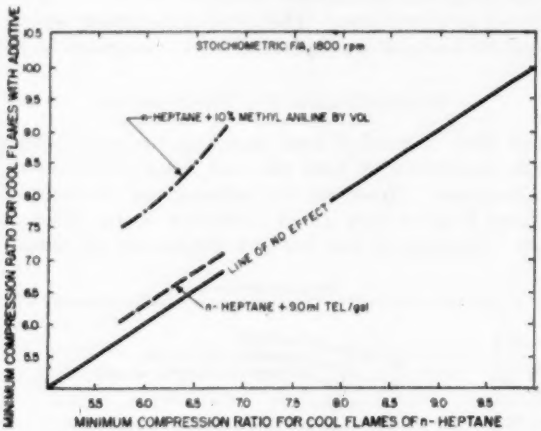
STOICHIOMETRIC F/A, 1800 rpm
n-HEPTANE + 10% METHYL ANILINE BY VOL
LINE OF NO EFFECT
n- HEPTANE + 9.0ml TEL/gal
MINIMUM COMPRESSION RATIO FOR COOL FLAMES WITH ADDITIVE
MINIMUM COMPRESSION RATIO FOR COOL FLAMES OF n- HEPTANE

FIG 2

EFFECT OF ANTIKNOCK COMPOUNDS ON THE LOWER COOL FLAME LIMIT OF *N*-HEPTANE

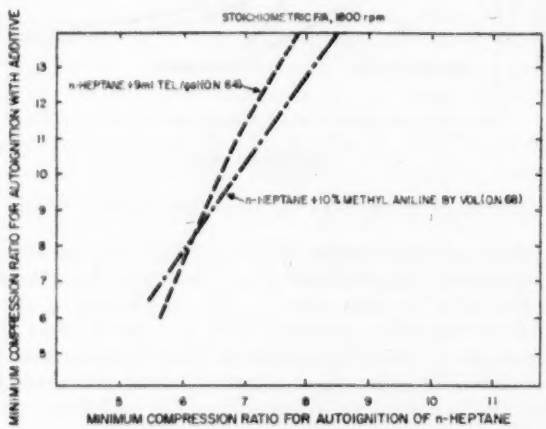
STOICHIOMETRIC F/A, 1800 rpm
n-HEPTANE +9ml TEL/gal(ON 84)
n-HEPTANE +10% METHYL ANILINE BY VOL(ON 68)
MINIMUM COMPRESSION RATIO FOR AUTOIGNITION WITH ADDITIVE
MINIMUM COMPRESSION RATIO FOR AUTOIGNITION OF n-HEPTANE

FIG 3

EFFECT OF ANTIKNOCK COMPOUNDS ON THE AUTO-IGNITION LIMIT OF *N*-HEPTANE

per gallon in extending the auto-ignition limit of *n*-heptane under relatively mild conditions. Under more severe conditions tetraethyl lead appears to become increasingly more effective, and at compression ratios above 8·5 : 1 it is actually more effective than methyl aniline in suppressing the auto-ignition of *n*-heptane.

The effects of these two antiknocks indicate that the auto-ignition of *n*-heptane can be delayed either by suppressing cool flame formation (as with methyl aniline) or by prolonging the cool flame process (as with tetraethyl lead). The auto-ignition resistance of pure hydrocarbons which exhibit

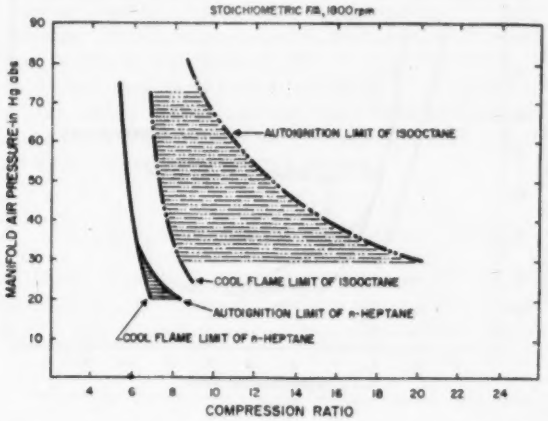

FIG 4

COOL FLAME AND AUTO-IGNITION LIMITS

two-stage ignition can be the result of either late cool flame formation, a prolonged cool flame process, or both of these factors. *iso*-Octane, for example, is not only resistant to cool flame formation, but its oxidation process is also such that an exceptionally long period is required for the cool flames to pass into auto-ignition. This effect is shown in Fig 4.

EFFECTS OF DI-*tert*-BUTYL PEROXIDE—A PEO-KNOCK

Peroxides are generally recognized as being important intermediate products in the precombustion reactions of hydrocarbons which exhibit two-stage oxidation. Experimental evidence has indicated that hydrogen peroxide, and various organic peroxide compounds are present during the early stages of oxidation. It has also been suggested that peroxides are intimately associated with knock, and recently it has been found that tetraethyl lead markedly reduces the concentration of peroxides in gases sampled from a firing engine.[9]

In a motored engine peroxide oxygen produces a considerable acceleration in the chemical reactions leading to auto-ignition. This is shown in Fig 5, which illustrates the effect of the presence of 5 mol per cent di-*tert*-butyl peroxide on the auto-ignition limit of *n*-heptane. The cool flame limit of this fuel is not indicated in Fig 5 because the di-*tert*-butyl peroxide promoted the oxidation to such an extent that the cool flames passed into auto-ignition almost immediately following their initiation, and it was not possible to differentiate between the cool flame and auto-ignition limits. Peroxides formed through precombustion reactions may exert a similar

accelerating effect on the combustion of *n*-heptane and other hydrocarbons in a fired engine; however, the effect is probably very much less because of their lower concentration.

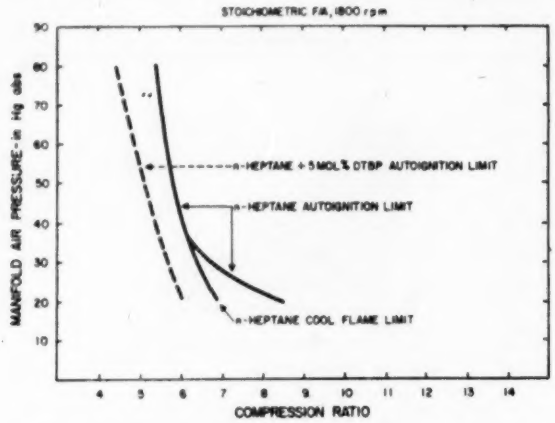
STOICHIOMETRIC F/A, 1800 r.p.m.
MANIFOLD AIR PRESSURE - in Hg abs
n-HEPTANE + 5 MOL % DTBP AUTOIGNITION LIMIT
n-HEPTANE AUTOIGNITION LIMIT
n-HEPTANE COOL FLAME LIMIT
COMPRESSION RATIO

FIG 5

EFFECT OF DI-*TERT*-BUTYL PEROXIDE ON *N*-HEPTANE IGNITION

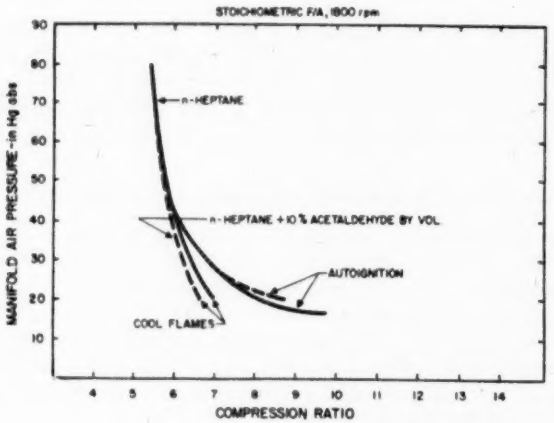

FIG 6

EFFECT OF ACETALDEHYDE ON *N*-HEPTANE PRECOMBUSTION REACTIONS

EFFECTS OF ACETALDEHYDE

Acetaldehyde also is one of the products formed by the precombustion reactions of many paraffins; it has been suspected frequently of being a precursor of knock. Pure acetaldehyde is much more reactive than *n*-heptane, and King and Durand [10] have demonstrated that it can be used

as an engine fuel only if low jacket temperatures and other mild conditions are employed. Knock tests [9-11] with gasolines containing large concentrations of acetaldehyde have indicated that it does not promote knock. Other experiments have indicated that acetaldehyde does promote knock when present in benzene.[9]

Motored engine studies with *n*-heptane containing 10 per cent acetaldehyde by volume (Fig 6) have revealed that acetaldehyde has no great effect on either the lower cool flame or the auto-ignition limits of *n*-heptane. Although cool flames may appear more readily when acetaldehyde is present, the effect is very small, and as the indicated differences in the lower cool flame limits are of the same order as the reproducibility of the data, it is possible that these differences may not be real.

Sulphur-tetraethyl Lead Antagonism

Small quantities of sulphur compounds, such as are usually present in commercial gasolines, can markedly reduce the antiknock effectiveness of tetraethyl lead. The extent of this antagonistic action has been found by

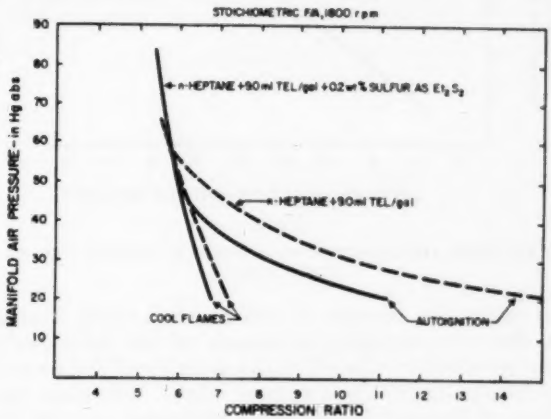

Fig 7

ANTAGONISTIC EFFECT OF SULPHUR ON TETRAETHYL LEAD

Livingston [12] to vary with the type and concentration of the sulphur compound and frequently to be such that over 60 per cent of the tetraethyl lead is rendered ineffective.

Disulphides are among the more antagonistic sulphur compounds; the presence of 0·2 weight per cent of diethyl disulphide in *n*-heptane containing 9 ml of tetraethyl lead per gallon has been found to reduce the octane number from 64 to 36. Fig 7 illustrates this antagonism by showing that lower compression ratios are required for both cool flame radiation and auto-ignition of *n*-heptane with 9 ml tetraethyl lead per gallon when disulphide sulphur is present.

Auto-ignition and Knock

The increased knocking tendency and the reduction in auto-ignition resistance which resulted from the addition of diethyl disulphide suggests, as do the effects which other fuel additives have been found to produce, that a correlation exists between auto-ignition in a motored engine and knock in a firing engine. In order to express the relationship between auto-ignition and knock on a more quantitative basis, the compression ratios required for auto-ignition in a motored engine operating at a manifold air pressure of 30 inches of Hg abs were plotted (Fig 8) against the standard

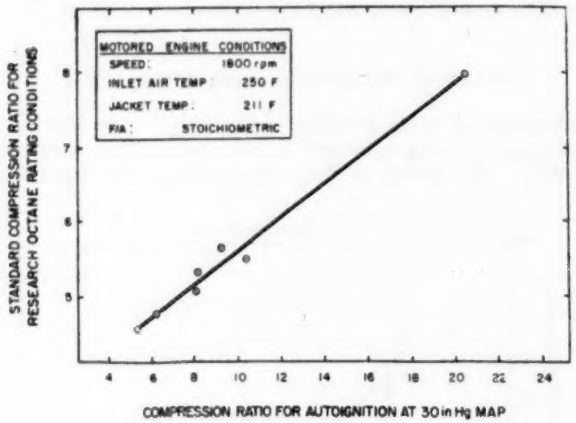

Fig 8

RELATION BETWEEN AUTO-IGNITION IN A MOTORED ENGINE AND KNOCK IN A FIRED ENGINE

compression ratios for research octane number rating conditions. This plot shows that the knocking tendencies of the fuels studied in this investigation are directly related to the severity of the conditions which are required for auto-ignition in a motored engine. Therefore, this investigation, like that of Pastell,[5] has provided additional support for the auto-ignition theory of knock and has demonstrated the decisive role that pre-combustion reactions have in determining the knocking characteristics of motor fuels.

Conclusions

The principal conclusions drawn from this investigation are :—

1. The motored engine is an excellent tool for the study of hydrocarbon oxidation because it permits a study of all phases of precombustion reactions and of the manner in which they are influenced by different fuel additives.

2. Tetraethyl lead does not appreciably influence the conditions required to obtain cool flames with *n*-heptane, but increases considerably the time required for cool flames to pass into auto-ignition, and as a consequence it

produces a substantial broadening of the range of physical conditions under which cool flames can exist.

3. Methyl aniline markedly retards the pre-cool flame reactions of *n*-heptane.

4. Di-*tert*-butyl peroxide greatly accelerates the cool flame reactions of *n*-heptane.

5. Acetaldehyde is not a primary cause of *n*-heptane knock.

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DETERGENCY OF CARBON BLACK

PART IV. INFLUENCE OF THE LIQUID PHASE ON THE SETTLING BEHAVIOUR OF DISPERSIONS

By F. H. Garner, O.B.E. (Fellow),* M. F. Mohtadi (Student Member),* and C. W. Nutt *

Summary

The rates of settling of carbon black (Micronex) have been investigated in a number of pure organic liquids and in solutions of additives. The detergent efficiency is measured up by the rate of settling of the dispersion, and in confirmation of the previous work, it is shown that the detergent efficiency of an additive can be correlated with its degree of adsorption on the carbon particles. The introduction of polar groups such as $-NO_2$, $-NH_2$, and halogens into the additive increases the detergent properties. The detergent efficiency of an additive in an organic liquid is not related to its efficiency in an aqueous solution. For a wide range of pure liquids and liquid mixtures the detergent efficiency can be correlated with the product of the density of liquid and its surface tension. It is also noted that many of the liquids of high detergent power also possess high dielectric constants.

Introduction

In earlier papers [1, 2, 3] it has been shown that the rate of settling of a carbon black (Micronex) suspension in a toluene solution of an additive may be employed to measure the detergent efficiency of the additive. The liquid phase has the ability, particularly in the presence of additives, to break down the large aggregates into smaller aggregates or possibly even primary particles, and thus the rate of settling is reduced. The rate of settling of Micronex suspensions is related to the degree of adsorption of the additive on the surfaces of carbon particles.

In this paper an investigation is made of the settling behaviour of Micronex suspensions in a number of pure organic liquids, of mixed organic liquids, and of solutions of acids, soaps, large-molecule surface-active agents, and commercial additives. The rates of settling measurements have been used to show that the detergent efficiency is related to the nature and composition of the dispersing liquid.

Experimental

The methods used for the preparation of the 25-ml samples of suspension, the determination of the adsorption of the surface-active agents, and the settling rates of the dispersions were those employed and reported in previous papers.[1, 2, 3]

* Department of Chemical Engineering, University of Birmingham.

Pure Organic Liquids

The liquids investigated included a wide range of compounds and are listed below :—

1. Toluene.
2. Benzene.
3. Methyl *cyclo*hexane.
4. Heptane.
5. Carbon tetrachloride.
6. Amyl alcohol.
7. Nitrobenzene.
8. Aniline.
9. Methyl propyl ketone.
10. Ethyl bromide.
11. Carbon disulphide.
12. Chloroform.
13. Benzaldehyde.
14. Bromobenzene.
15. *o*-Bromotoluene.
16. *o*-Chlorotoluene.
17. Mono-chlorobenzene.
18. *o*-Nitrotoluene.
19. White spirit.

The samples used were of high purity, usually of AR grade.

The rates of settling of Micronex suspensions in the above liquids are indicated by the settling curves of Figs 1 (*a*) and 1 (*b*), where the term " settled volume " signifies that column of clear solution above the settling suspension.

The results show that heptane, methyl *cyclo*hexane, white spirit, and toluene have high settled volumes and consequently relatively poor dispersing power. On the other hand, aniline, nitrobenzene, bromobenzene, bromotoluene, benzaldehyde, and *o*-nitrotoluene have dispersing properties so strong that no settling of Micronex occurs in these liquids. Carbon tetrachloride and others are intermediate between the extremes.

Organic Liquid Mixtures

To examine further certain correlations obtained from the study of dispersions in pure organic liquids, and to assess the relative effect of a liquid on the detergent properties of binary mixtures, the following organic liquid mixtures were investigated :—

1. 75 per cent toluene + 25 per cent nitrobenzene.
2. 75 per cent toluene + 25 per cent aniline.
3. 75 per cent toluene + 25 per cent carbon tetrachloride.
4. 75 per cent heptane + 25 per cent nitrobenzene.
5. 75 per cent heptane + 25 per cent carbon tetrachloride.
6. 75 per cent methyl *cyclo*hexane + 25 per cent nitrobenzene.
7. 75 per cent methyl *cyclo*hexane + 25 per cent carbon tetrachloride.
8. 50 per cent toluene + 50 per cent nitrobenzene.
9. 50 per cent toluene + 50 per cent aniline.
10. 50 per cent toluene + 50 per cent carbon tetrachloride.
11. 50 per cent heptane + 50 per cent nitrobenzene.
12. 50 per cent heptane + 50 per cent carbon tetrachloride.
13. 50 per cent methyl *cyclo*hexane + 50 per cent nitrobenzene.
14. 50 per cent methyl *cyclo*hexane + 50 per cent carbon tetrachloride.
15. 25 per cent toluene + 75 per cent nitrobenzene.
16. 25 per cent toluene + 75 per cent aniline.
17. 25 per cent toluene + 75 per cent carbon tetrachloride.
18. 25 per cent heptane + 75 per cent nitrobenzene.
19. 25 per cent heptane + 75 per cent carbon tetrachloride.
20. 25 per cent methyl *cyclo*hexane + 75 per cent nitrobenzene.
21. 25 per cent methyl *cyclo*hexane + 75 per cent carbon tetrachloride.

(All percentages are by volume.)

Fig. 2 shows the settling curves for Micronex suspensions in some of these organic liquid mixtures. No settling occurred in mixture numbers 4,

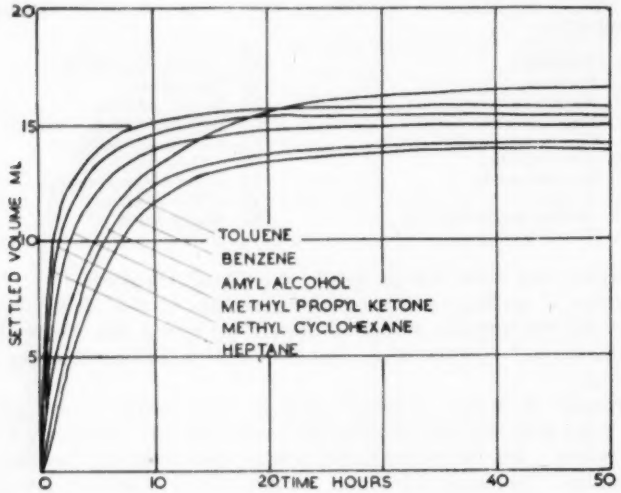
SETTLED VOLUME ML
20
15
10
5
0
10
20
TIME HOURS
40
50
TOLUENE
BENZENE
AMYL ALCOHOL
METHYL PROPYL KETONE
METHYL CYCLOHEXANE
HEPTANE

FIG 1 (*a*)

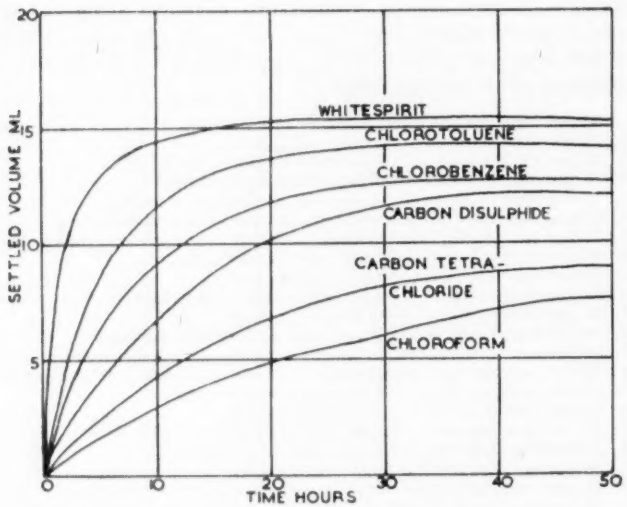
SETTLED VOLUME ML
20
15
10
5
0
10
20
30
40
50
TIME HOURS
WHITESPIRIT
CHLOROTOLUENE
CHLOROBENZENE
CARBON DISULPHIDE
CARBON TETRA-
CHLORIDE
CHLOROFORM

FIG 1 (*b*)

9, 11, 13, 15, 16, 18, 20. The presence of aniline, nitrobenzene, or carbon tetrachloride caused a marked improvement in the suspendibility of Micronex in toluene, heptane, and methyl *cyclo*hexane.

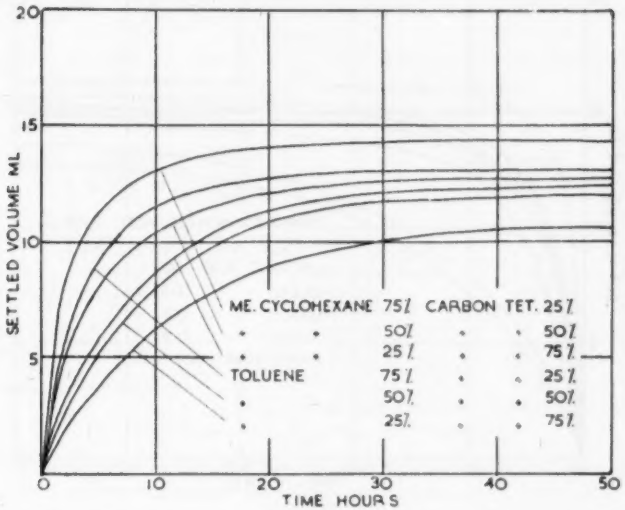

FIG 2

Acid and Soap Additives

The following compounds were investigated :—

(i) *Acids*

1. Oleic acid.
2. Linoleic acid.
3. Stearic acid.
4. Lauric acid.
5. Benzoic acid.
6. Succinic acid.
7. Oxalic acid.
8. Tannic acid.
9. Naphthol disulphonic acid.
10. Palmitic acid.
11. Dibromolinoleic acid.
12. Naphthenic acid.

The acids were in some cases pure AR reagents (*i.e.*, oxalic, succinic, and benzoic acids), others were laboratory reagents. Dibromolinoleic acid was prepared in the laboratory by the slow bromination of linoleic acid at 0° C.

(ii) *Soaps*

1. Calcium oleate.
2. Sodium oleate.
3. Lead oleate.
4. Ethyl oleate.
5. Calcium stearate.
6. Nickel stearate.
7. Sodium stearate.
8. Ethyl stearate.
9. Calcium linoleate.
10. Sodium linoleate.
11. Calcium naphthenate.
12. Barium naphthenate.
13. Calcium oxalate.
14. Calcium laurate.

The calcium soaps were prepared by the substitution in the corresponding sodium soaps.[4] Other soaps used were obtained as ordinary laboratory reagents.

Some of the above acids and soaps were only slightly soluble in toluene. Such soaps were dissolved in hot toluene, but on cooling a viscous and colloid-like solution was formed which slowly deposited a gel. Thus the

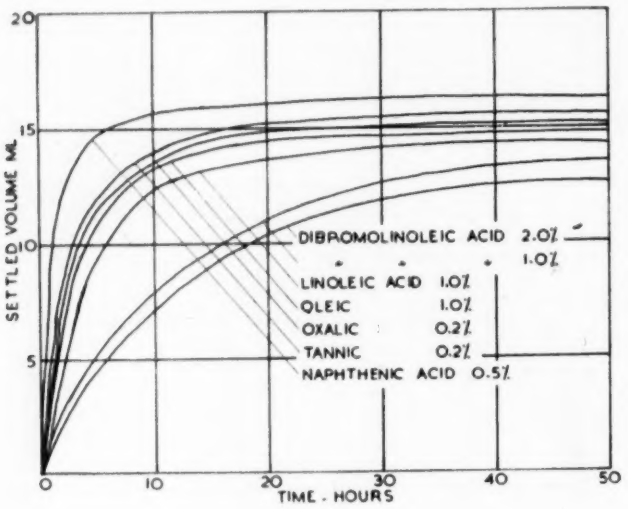

Fig 3

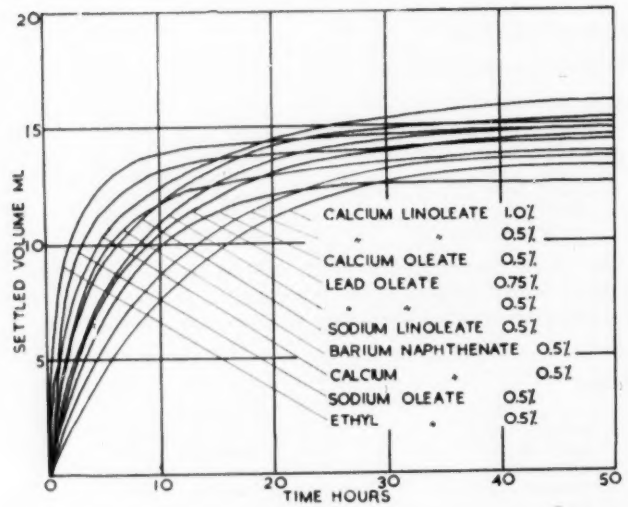

Fig 4

concentration of the solution was found to alter from day to day, and was, therefore, frequently checked using the analytical method previously described.[4]

Figs 3 and 4 show the results of the settling experiments in acid and soap solutions.

Measurements were also made of the adsorption isotherms of some of these soaps and acids. The method employed was that described in an earlier paper,[1] and some of the results are tabulated in Table I.

TABLE I

Acids			Soaps		
	Concn g/100 ml	Micro-moles adsorbed		Concn g/100 ml	Micro-moles adsorbed by 0·5 g Micronex
Oleic acid .	0·5 and 1·5	Negligible	Calcium oleate .	0·5	36·0
Linoleic acid .	0·5 and 1·0	,,	Calcium oleate .	1·0	39·0
Naphthenic acid	0·25		Calcium . .	0·25	44·3
Stearic acid .	0·25	,,	Linoleate . .	1·0	51·7
Lauric acid .	0·25	,,	Calcium naph-		
Palmitic acid .	0·25	,,	thenate . .	0·25	22·0
Naphthol disul-			Barium naph-		
phonic acid .	0·25 and 0·5	,,	thenate . .	0·50	20·0
			Calcium stearate	0·25	26·0

From a comparison of Fig 3 with Fig 1 it will be observed that but for one exception (dibromolinoleic acid) the acids have had very little effect on the stability of suspensions. Soaps, on the other hand, have effected a notable improvement in the rates of settling. Measurements of the amount of adsorption of the additives on carbon revealed that insignificant amounts of acids were adsorbed, whereas soaps were adsorbed in appreciable quantities. The adsorption isotherms were very similar to those shown in a previous paper.[3] These all revealed a saturation effect at approximately 1·5 to 2·0 per cent concentration of soaps, coinciding with the concentration required to achieve optimum stability of the suspension as indicated by the settling behaviour.

Large-molecule Surface-active Compounds

Large-molecule surface-active compounds such as alkyl sulphonates and polyoxyethylenes are used extensively as washing detergents in the textile industries and in the household. The stabilizing effect of a few of these compounds on suspensions of Micronex in toluene was investigated. The compounds used included :—

1. Polyoxyethylene benzyl alcohol.
2. Polyoxyethylene surbitol stearate.
3. Polyoxyethylene penta stearate (G 2151).
4. Polyoxyethylene stearate (Estax 22.H.4).
5. Polawax.
6. Sodium 6-chloro, 5-nitrotoluene sulphonate.
7. Sodium 5-nitro, 6-chlorotoluene sulphonate.

All the compounds were obtained from the Carbide and Carbon Chemicals Corporation of U.S.A.

Compounds (1) to (5) were found to be easily soluble in toluene, and their

solutions (up to 5 per cent) were stable over a long period. The two sulphonate solutions were unstable, forming a gel, which caused difficulty in estimation of their true concentration. Most suspensions were relatively unstable, having high initial rates of settling and low final sediment volumes.

Commercial Lubricating-oil Additives

For comparison of the dispersing power, the settling behaviour of Micronex suspensions in toluene solutions of four commercial lubricating-oil additives (called additive B, C, D, and E) was investigated. The oils

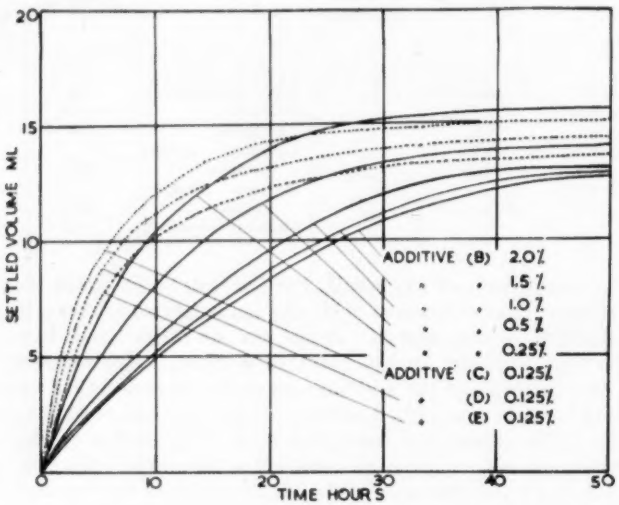

FIG 5

examined were highly coloured viscous liquids. They were easily soluble in toluene and had a specific gravity of approximately 1·0.

Suspensions made in solutions of additives C, D, and E were found to be extremely stable. No settling occurred when the additive concentration in the solution was above 0·5 per cent by volume. By reducing the concentration to 0·25 per cent and further to 0·125 per cent an extremely slow and only partial settling of the suspended carbon particles was observed during 48 hr. To detect such partial sedimentation it was necessary to view the settling tube against an intense source of light. This showed that the bulk of the carbon had sedimented to an apparent final sediment volume of 9 to 10 ml. The solution above the sediment, however, contained large numbers of particles of extremely small size.

The rate-of-settling curves for additive B samples are plotted in Fig 5, and they show the very slow rates of settling of these dispersions. The partial settling of the samples in additives C, D, and E are shown in dotted lines. Adsorption measurements for these additives indicated that the amount of oil adsorbed on carbon black increased with increasing concen-

tration of the oil until a saturation point (2·0 per cent for additive B) was reached. The results were similar to those found and reported in a previous paper.[3]

Comparison of Toluene and Heptane as Dispersing Agents

To investigate the influence of the solvent on the detergent properties of surface-active additives, measurements were made of the settling rates of dispersions in toluene and heptane solutions of the following compounds :

1. Linoleic acid.
2. Dibromolinoleic acid.
3. Barium naphthenate.
4. Lithium stearate.

The results are shown in Fig 6, from which it can be clearly observed that

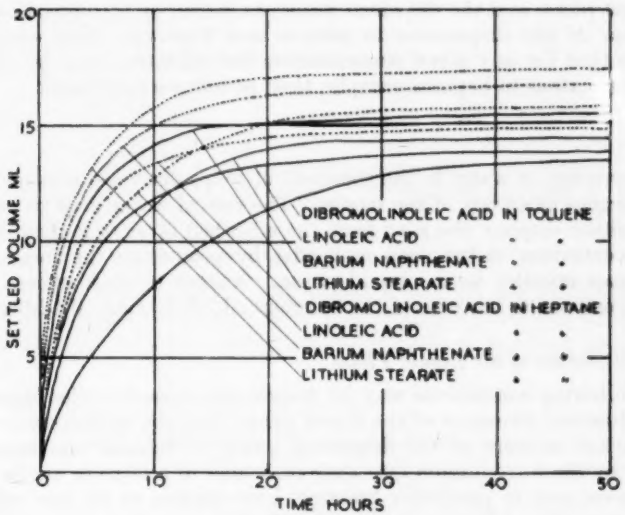

Fig 6

the heptane suspensions were less stable than the corresponding toluene samples. This is particularly marked in the case of dibromolinoleic acid dispersions.

Effect of Small Amounts of Water

The rate of settling of dispersions in solutions of additive B, calcium oleate, and calcium naphthenate in toluene containing a known volume of dissolved water was determined. The amount of water added (0·03 to 0·5 ml per 25 ml solution) constituted a small fraction only of the total volume. It was either mixed with the solution prior to the dispersion of Micronex (using ultrasonic waves for mixing) or added to the carbon black.

No appreciable change in the settling behaviour of dispersions in additive B was effected by the added water. In the case of soaps the suspension

stability was slightly reduced. The samples sedimented with relatively fast rates and occupied smaller final sediment volumes.

Discussion and Conclusions

The Adsorption of Surface-active Additives

The comparison of the settling rates of dispersions shows that the soaps in toluene solutions have a considerably greater detergent power than the acids. Further, the soaps are adsorbed on carbon black to a much larger extent than acids. The maximum detergent efficiency for a given additive is attained at the concentration for which its adsorption is greatest. The solvent itself influences the distribution of surface-active additives between the liquid phase and the dispersed phase, as shown by a comparison of the behaviour of the dispersions in toluene and heptane. This comparison suggests that for any given concentration the additives may be adsorbed to a lesser extent in heptane samples than in toluene dispersions.

Water

The presence of water in the solutions of soaps decreases to some extent the detergent efficiency of the media. The role of water may be twofold : it may either displace the soap from the adsorbed layer or it may decrease the concentration of the soap molecules by promoting the formation of water–soap micelles in the liquid phase. Neither of these effects appear to be significant in the case of the commercial lubricating-oil additive B.

Chemical Nature of the Liquid Phase

The following conclusions may be drawn with regard to the importance of the chemical structure of the liquid phase (and the additives) :—

A marked increase in the dispersing power of toluene and benzene is effected by the introduction into their molecules of a nitro or amine group.

Halogens, and in particular bromine, have proved to be very effective. The bromination of linoleic acid has resulted in a product (dibromolinoleic acid) with a much improved dispersing power. The suspensions in other brominated compounds did not settle during the test period of 48 hr.

The presence of unsaturated bonds also appears to be an effective factor. Calcium oleate (one double bond) is adsorbed on carbon black to a much greater extent than calcium naphthenate (saturated molecule), but considerably less than calcium linoleate (two double bonds).[3] In these cases the adsorption has been accompanied by a corresponding improvement in the suspension stability. Analogous results have been reported by Damerell and Mattson [5] for adsorption of various additive compounds in carbon–xylene systems.

It is the presence of metal radicals which probably provides the basic difference between the suspending power of soaps and the corresponding acids. Barium and calcium appear to be more effective than lead, sodium, nickel, and organic radicals (*i.e.*, ethyl radical).

The size of the molecule of an additive does not seem to play an important part in the detergent properties.

The efficiency of a detergent for dispersing carbon in organic liquids bears no relation to its efficiency when used in aqueous solution. Thus the strong " aqueous " detergents derived from polyoxyethylenes are much less efficient than the single halogenated compounds which would have negligible detergent property in aqueous systems. This is to be expected in view of the great difference in the aqueous and organic liquid media.

Physical Properties of the Liquid Phase

It may be shown, and is further discussed in Part VI, that the main part of the sedimentation curve for a micronex suspension follows the equation :—

$$\log [(V_t - V_\infty)/(V_0 - V_\infty)] = Kt$$

where V_t is the volume of the suspension at time t;
V_0 is the initial volume of suspension;
V_∞ is the final volume of suspension at $t = \infty$;
t is time;
and K is a constant.

Thus the values of K and V_∞ can define adequately the course of the main part of the sedimentation curve for any suspension and therefore describe the detergent properties of the liquid. The numerical value of K can be determined from a plot of :—

$$\log [(V_t - V_\infty)/(V_0 - V_\infty)] \textit{ vs } \text{time}$$

for a given suspension sample. In Tables II and III are listed the values of K thus derived for suspensions in pure organic liquids and some liquid-mixture systems, together with the more important physical properties of

TABLE II

Some of the Physical Properties of Organic Liquids *

Organic liquids	Density	Density difference between Micronex and liquids	Surface tension	Parachor	Electrical conductivity, 0° C	Dielectric constant, 20° C	Density × surface tension	K	Viscosity " centipoise," 20° C
Heptane	0·69	1·11	20·4	307·0	$0·9 \times 10^{-12}$	1·97	14·2	3·0	0·43
Methylcyclohexane	0·765	1·04	23·2	279·7	—	2·01	17·6	2·0	0·45
Amylalcohol	0·812	0·988	23·0	249·0	5×10^{-8}	16·0	18·8	2·0	4·3
Methylpropylketone	0·809	0·991	29·15	239·0	1×10^{-8}	16·0	20·1	1·7	0·46
White spirit	0·823	0·977	26·1	—	—	—	21·2	1·9	—
Toluene	0·864	0·936	27·9	246·0	$1·9 \times 10^{-14}$	2·3	24·4	1·6	0·61
Benzene	0·87	0·93	28·9	207·1	—	2·8	25·7	1·3	0·67
White oil (light)	0·859	0·941	32·0	—	—	—	—	—	—
White oil (heavy)	0·885	0·915	32·0	—	—	—	—	—	—
Ethyl bromide	1·45	0·35	24·3	163·1	2×10^{-9}	9·45	35·2	NS	0·39
o-Chlorotoluene	1·08	0·72	28·1	282·5	—	4·7	29·8	1·1	1·08
Carbon disulphide	1·26	0·54	31·4	148·2	$7·8 \times 10^{-6}$	—	38·2	0·66	0·36
mono-Chlorobenzene	1·15	0·65	33·8	243·8	—	5·9	39·1	0·50	0·90
Carbon tetrachloride	1·58	0·22	26·7	220·0	4×10^{-13}	2·24	41·2	0·46	1·05
Benzaldehyde	1·043	0·76	42·0	255·1	17×10^{-8}	18·0	42·7	NS	—
Chloroform	1·51	0·29	27·13	183·3	2×10^{-9}	5·05	41·4	0·30	0·57
Aniline	1·026	0·78	42·6	236·7	9×10^{-9}	7·2	43·5	NS	4·5
o-Bromotoluene	1·42	0·38	35·8	297·0	—	5·4	51·0	NS	1·45
Nitrobenzene	1·2	0·60	43·4	242·5	5×10^{-9}	35·7	53·0	NS	2·15
Bromobenzene	1·52	0·28	36·3	258·0	2×10^{-11}	5·4	55·0	NS	1·56

NS = No settling observed.

the liquids.* In Table II the liquids have been arranged in the order of the increasing dispersing power, as revealed by present experimental results. The Parachor values have been calculated from "Parachor equivalents" of chemical groupings given in the literature.[6] In Table III are data for binary mixtures of pure liquids which have been determined in the laboratory. The surface-tension measurements were made with a "du Nouy"-type tensiometer.

TABLE III

Some of the Physical Properties of Organic Liquid Mixtures

Liquid mixtures	Density at 20° C	Surface tension, 20° C	Density × surface tension	K
Nitrobenzene (25%) + toluene	0·945	29·0	27·4	—
" " + methylcyclohexane	0·873	23·5	20·6	NS
" " + heptane	0·815	21·7	17·8	—
" (50%) + toluene	1·034	31·5	32·7	—
" " + methylcyclohexane	0·987	26·2	25·7	NS
" " + heptane	0·964	23·4	22·5	NS
" (75%) + toluene	1·114	36·0	41·0	NS
" " + methylcyclohexane	1·092	30·5	33·3	NS
" " + heptane	1·074	26·7	28·6	NS
CI_4 (25%) + toluene	1·03	26·5	27·2	1·30
" " + methylcyclohexane	0·973	23·5	23·5	1·75
" " + heptane	0·916	21·5	21·5	1·82
" (50%) + toluene	1·28	28·1	36·0	0·62
" " + methylcyclohexane	1·18	25·1	29·3	0·84
" " + heptane	1·14	23·0	26·2	1·45
" (75%) + toluene	1·44	26·5	38·1	0·55
" " + methylcyclohexane	1·38	25·0	34·6	0·78
" " + heptane	1·35	24·9	34·0	0·80
Aniline (25%) + toluene	0·912	29·5	26·8	NS
" (50%) + "	0·945	31·2	29·3	NS
" (75%) + "	0·979	35·1	34·4	NS
Toluene (50%) + heptane	0·775	23·5	18·23	—
" " + methylcyclohexane	0·794	24·8	19·4	—

NS = No settling observed.

From an examination of the data of Table II it will be observed that there is no simple relation between the suspension stability and the liquid density, or the density difference between the carbon black and the liquids. The surface tension of the liquid is closely related to the internal pressure, and might therefore be expected to bear a direct correlation with the dispersing power of the liquid, but there appears to be no simple relation between these properties. However, it may be that it is the surface tension of the "suspension" which is the important factor. It is known that very gradual changes in the surface tension of a dispersion may occur and the suspension may exhibit a much higher surface tension than that of pure liquid. Such high surface tensions (of the order of several hundred dynes/centimetre) have been observed by Antonoff [7] in thick suspensions of carbon black in olive oil and lauryl alcohol.

* From "International Critical Tables," McGraw-Hill Co., N.Y.

The electrical conductivity of the pure liquids does not show any simple correlation with detergent efficiency, but it is apparent that with one exception, all liquids of strong dispersing power possess high dielectric

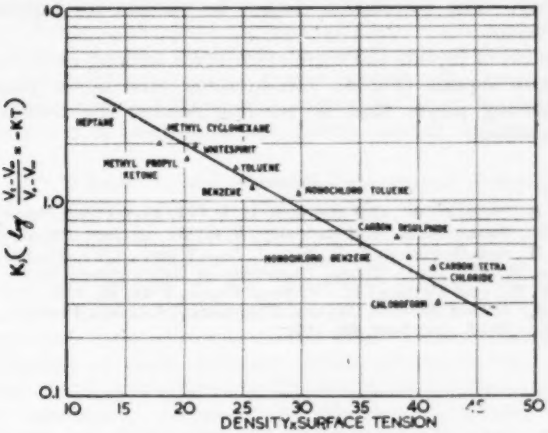

FIG 7

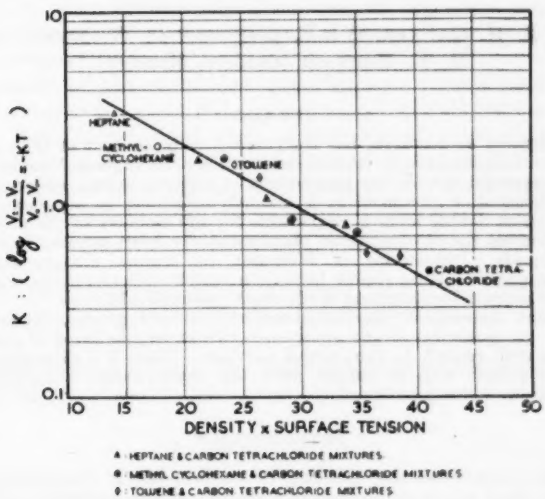
10
1.0
0.1
10 15 20 25 30 35 40 45 50
DENSITY x SURFACE TENSION
HEPTANE
METHYL - O - CYCLOHEXANE
TOLUENE
CARBON TETRA CHLORIDE
A : HEPTANE & CARBON TETRACHLORIDE MIXTURES
⊕ : METHYL CYCLOHEXANE & CARBON TETRACHLORIDE MIXTURES
0 : TOLUENE & CARBON TETRACHLORIDE MIXTURES

FIG 8

constants. It can be assumed generally that the liquids of high dielectric constant have good detergent efficiency.

For a number of liquids the Parachor bears some relation to the detergent properties of the liquids. A more complete correlation, however, may be obtained using the product of: density × surface tension of the liquids

($\rho \times \gamma$)—Table II (col. 8), indicates that the increase in the numerical value of $\rho \times \gamma$ has always been accompanied by the increase in the detergent efficiency for various organic liquids. Thus a plot of log K *vs* $\rho \times \gamma$ (Fig 7) shows a linear relation for those liquids in which settling occurs under gravity. The correlation cannot be verified for liquids in which no settling occurs.

For mixtures of liquids the correlation holds as long as the settling can occur in both liquids (Fig 8). If, however, one liquid possesses such strong dispersing power that no settling occurs, the correlation again cannot be verified.

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[3] Garner, F. H., and Baddeley, A. R. W. *J. Inst. Petrol.*, 1951, **37**, 426.
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PART V. ELECTROKINETIC PHENOMENA IN MICRONEX DISPERSIONS IN ORGANIC LIQUID MEDIA

By F. H. GARNER, O.B.E. (Fellow),* C. W. NUTT,* and M. F. MOHTADI (Student Member) *

SUMMARY

An electrical double layer has been shown to be present at the surface of particles of carbon black (Micronex) dispersed in organic liquids. These disperse systems exhibit the phenomena of electrophoresis, electro-osmosis, and sedimentation potential in a manner analogous to aqueous systems. The electrical double layer is responsible for the stability of the dispersions, and in general the electrokinetic phenomena are more marked in the more polar liquids. Dispersions of Micronex in organic liquids containing detergent additives show similar behaviour, and the electrokinetic phenomena are more marked for solutions of the most powerful additives.

Micronex dispersions may be classified into two groups, depending on whether the potential of the outer shell of the double layer is positive or negative with respect to the carbon particles. Thus if a dispersion of one group is mixed with a sample from the other group, coagulation and precipitation will result.

INTRODUCTION

IN previous papers on the settling of carbon-black dispersions [1-4] it has been shown that the final sediment volume of 0·5 g of Micronex dispersed in 25 ml of organic liquid is of the order of 10 ml. Since the apparent specific gravity of Micronex is approximately 1·8, this sediment volume is many times greater than the volume of the dry Micronex. The large increase in the sediment volume suggests the existence of strong forces of interaction between the particles in the dispersion. These forces may arise

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from the formation of a rigid chain or scaffolding structure of particles, or alternatively they may arise from a thick adsorbed layer of liquid molecules at the carbon–liquid interface. In the latter case an eightfold increase in the final sediment volume can take place when the thickness of the adsorbed layer is equal to the radius of the particles. For the Micronex used it was known that the average particle size was of the order of 50 mμ. It is necessary, therefore, to postulate an adsorbed layer at least 50 mμ thick or an electrical double layer at the interface. Such an electrical double layer around the surface of particles will give rise to such phenomena as electrophoresis, electro-osmosis, and sedimentation potential.

Damerell and Urbanic [6] were able to demonstrate electrophoresis in organosols of carbon in xylene in the presence of surface-active agents. They observed migration towards the positive electrode, indicating a negative charge on the carbon particles. The velocity of migration depended upon the nature of the dispersing media. Sols containing lecithin or sodium dioctyl sulphosuccinate showed migration at 110 volts, whereas sols containing soaps required 900 volts before migration could be detected. Dryness or slight moisture in the system did not effect the migration, and even under extremely dry conditions the organosols showed cataphoretic movement. Damerell and Mattson [5] extended the study of cataphoresis to calcium carbonate sols in xylene in the presence of a number of additives. The migration of the particles was towards the cathode, except when lecithin had been used as dispersing agent. With lecithin some particles migrated towards positive and some towards negative electrodes.

Electrophoresis of the suspended particles and electro-osmosis of the liquid phase increase the electrical conductivity of the dispersion. Forster and Mead [7] have investigated the conductivity of suspensions of R-40 carbon black in transformer and linseed oils in a rotational test cell. They showed that information regarding the shape and state of agglomeration of the suspended particles can be obtained from measurements of the dielectric constant and conductivity of the suspensions under different conditions of shear.

When particles surrounded by an electrical double layer are allowed to fall through a liquid, a sedimentation potential is set up, as shown by Dorn.[8] Stock [9] confirmed that for the movement of pure powdered quartz through organic liquids, the sedimentation potential (E) is given by Smoluchowsky's equation based on Helmholtz' theory of the double layer :—

$$E = \zeta DPR/4\pi\eta$$

where ζ is the zeta potential across the diffuse double layer at the interface;
D is the dielectric constant of the liquid;
P hydrostatic pressure difference;
η viscosity of liquid;
R specific resistivity.

More recently Quist and Washburn [10] have shown similar agreement with theory for inorganic powders in aqueous systems.

For aqueous systems these electrokinetic phenomena can be explained in terms of the theory of the electrical double layer at the interface, suggested by Helmholtz and later modified by the diffuse double-layer theory of Gouy and Freundlich.[11, 18] On this basis the electrophoretic mobility and the sedimentation potential may be related to the phase boundary potential and the zeta potential (the potential across the diffuse double layer). Generally, for the phase boundary potential difference in non-aqueous media, the rule put forward by Coehn [12] is of importance. This rule states that a substance of higher dielectric constant in contact with a substance of lower dielectric constant charges itself positively with regard to the latter. Coehn also found that the electrokinetic phenomena were more marked in organic liquids of high dielectric constant than in the liquids of low dielectric constant.

In recent years several attempts have been made to correlate the observations on the suspendibility of powders in detergent solutions with postulated changes in the zeta potential and the electrokinetic behaviour. Ray and Hutchinson [17] have correlated the suspendibility properties of dilute aqueous soap solutions for carbon black with the velocity of electrophoresis of particles. In more concentrated solution of detergents, however, Doscher's [13] measurements on systems of cellulose in solutions of cationic surface-active agents suggest that the suspendibility may also depend on whether the detergent can be adsorbed in ionized form.

In colloidal sols the two most important stabilizing factors appear to be :—

(1) the electric charges carried by the particles;
(2) the extent of solvation of the colloidal micelle.

Coagulation of sols can also take place when the dispersed particles are deprived of their charged layer or solvated ions.

The present work has been concerned with demonstrating the existence of the electrokinetic phenomena at the interface between dispersed carbon particles and organic liquids, and its relation to the detergent properties of the liquid. Electrophoresis and electro-osmosis have been demonstrated for carbon suspensions, the sign of the electrical charge on the particles being dependent on the nature of the liquid phase. The settling behaviour of the suspensions can be modified by an external e.m.f., and the existence of a sedimentation potential has been demonstrated in suspensions settling under gravity.

Experimental

Electrophoretic Behaviour of Micronex Dispersions

In a preliminary experiment the electrophoresis of carbon black (Micronex) dispersed in 0·25 per cent solution of a commercial additive (additive E) in toluene was demonstrated in a three-limbed U-tube. The tube was first half-filled with additive solution. Micronex suspension was then added slowly through the third limb, which was attached to the bottom of the U-bend, until each of the main side limbs was filled with the additive solution in its top half. The lower half of each limb and the U-bend now contained the dispersion. A potential difference of 240 volts D.C. was

applied between the two platinum-disk electrodes situated near the top of each limb and immersed in the additive solution. A gradual sharpening of the interface between the solution and the dispersion took place in both limbs. After 1 hr of application of the field the interface in the negative limb had descended approximately 2·3 mm, whilst that in the positive limb had ascended a similar distance. Reversal of the electric field reversed the direction of migration.

Thus carbon particles dispersed in solution of additive E were shown to have a negative charge, of such magnitude as to give rise to a cataphoretic velocity of 0·23 cm/hr in a field of 11·5 volts/cm or 0·02 cm/hr per unit field gradient.

In a U-tube entirely filled with carbon dispersion in a solution of additive E, application of the electric field results in settling of the dispersion in the limb with the negative electrode. This settling takes place in a similar way to the free settling of Micronex suspensions under gravity, *i.e.*, by the formation of a sharp interface between clear solution and dispersion and the downward movement of this interface. The rate of sedimentation depends on the voltage applied.

Using this method, the behaviour of a number of suspensions was investigated with fields in the range 40 to 250 volts/cm. It was found that the suspensions could be classified into two groups :—

A. Systems in which the disperse phase is negatively charged—

1. Toluene.
2. Heptane.
3. Methyl*cyclo*hexane.
4. Solutions of commercial additives B, C, D, and E.
5. Oleic acid in toluene.
6. Calcium naphthenate in toluene.

B. Systems in which the disperse phase is positively charged—

1. Nitrobenzene.
2. Nitrotoluene.
3. Bromobenzene.
4. Bromotoluene.
5. Chloroform.

Where settling did not occur under gravity, settling in one limb only could be induced by the application of the electric field. In those systems where settling occurs under gravity application of the field after gravitational settling had already begun gave a reduction of the settling rate in one limb and an increase of the rate in the other. Fig 1 shows a typical settling curve for the two limbs of a U-tube containing a dispersion in 2·0 per cent solution of linoleic acid. The electric field was applied after ½ hr.

In solutions of additive E, for a given electric field, increase of additive up to 4·0 per cent concentration resulted in an increase of the rate of settling in the negative limb. The size and shape of the electrodes, or the carbon concentration (within the limits 0·1 to 0·5 g Micronex in 50 ml of liquid) did not significantly affect the velocity of the electrophoretic migration. On removal of the electric field (after the complete settling of the dispersion) redispersion of the sediment occurred. The process of redispersion was, however, very slow, and required a period of 4 to 5 days. Reversal of the electric field or application of an external electrostatic charge near the interface readily increased the rate of such redispersion.

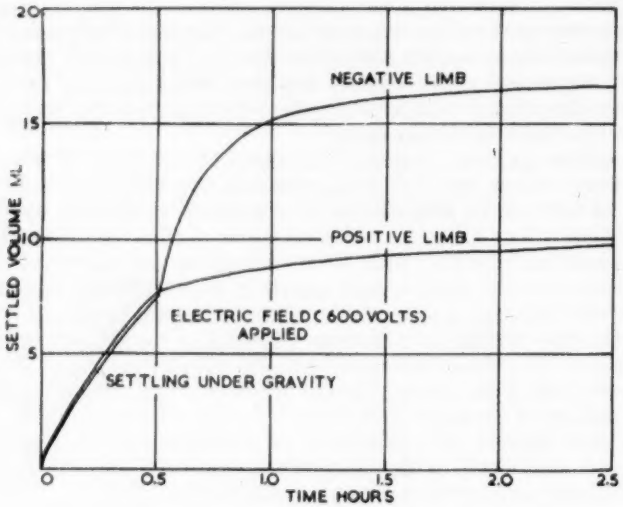
NEGATIVE LIMB
POSITIVE LIMB
ELECTRIC FIELD (600 VOLTS) APPLIED
SETTLING UNDER GRAVITY
SETTLED VOLUME ML
TIME HOURS
0
0.5
1.0
1.5
2.0
2.5
5
10
15
20

Fig 1

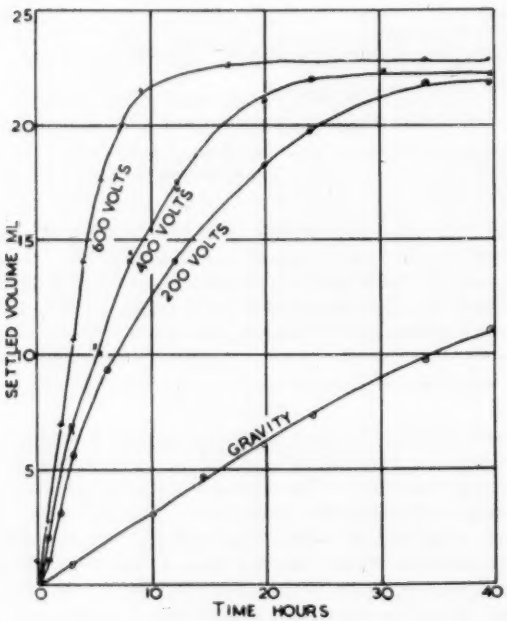
600 VOLTS
400 VOLTS
200 VOLTS
GRAVITY
SETTLED VOLUME ML
TIME HOURS
0
10
20
30
40
5
10
15
20
25

Fig 2

The results shown in Fig 1 indicate that the final sediment volume is influenced by the applied electric field. This was confirmed by experiments using a single vertical settling tube (1·4 cm internal diameter and

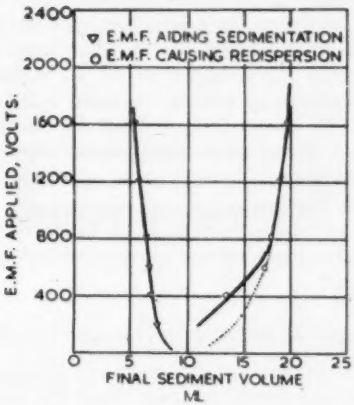

FIG 3

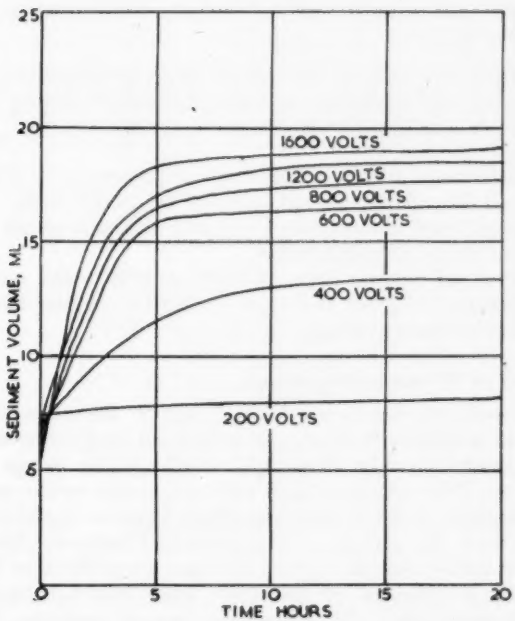

FIG 4

30 ml graduated capacity) with platinum electrodes inserted in top and bottom of the tube. Fig 2 indicates the settling curves for identical suspensions of Micronex in 3·0 per cent solution of commercial additive

B in toluene, under gravity and with various applied voltages. It shows that high potentials increase the rate of settling and decrease the final sediment volume. Fig 3 shows the variation of final sediment volumes with applied electric field for the same sample.

If a dispersion of Micronex in a solution of commercial additive E is forced to settle electrophoretically in a straight tube, reversal of the field will not completely cause the redispersion of the sediment. The interface moves slowly with a decreasing velocity to some value always less than the original volume of the dispersion, depending on the applied voltage. Fig 4 illustrates the form of such redispersion curves.* Similar results were obtained for Micronex dispersions in nitrobenzene, bromobenzene, *o*-nitrotoluene, and chloroform.

The failure of the sediment to redisperse fully to the initial dispersion volume (V_0) suggests that the forced settling process results in an irreversible packing of the sediment.

Behaviour of Suspensions in Mixtures of Groups (A) and (B)

The behaviour of suspensions in mixtures of groups (A) and (B) shows the importance of electric forces in controlling the final sediment volume of the dispersion. Suspensions prepared in 2·0 per cent solution of additive E and nitrobenzene were mixed in the following proportions (volume percentage) :—

(i) 25 per cent additive E—75 per cent nitrobenzene;
(ii) 50 per cent additive E—50 per cent nitrobenzene;
(iii) 75 per cent additive E—25 per cent nitrobenzene.

When allowed to stand for 72 hr the suspensions settled to final sediment volumes of : (i) 2·0 ml, (ii) 1·8 ml, (iii) 2·5 ml, respectively. This is in striking contrast to the non-settling behaviour of nitrobenzene suspensions and suspensions containing additive E.

Application of an electric field of 1000 or 2000 volts resulted in no significant changes in either the rate of settling or the final sediment volume of these mixture systems.

Electro-osmosis in Micronex Dispersions

The electro-osmotic behaviour of samples of Micronex dispersion in the commercial additives B, C, D, and E (2·0 per cent solution in toluene) was demonstrated in a tube of the type employed by Briggs *et al* [14] and shown in Fig 5. The tube was filled with suspension to the points A and A_1, and the additive solution added carefully until the liquid level reached the points B and B_1, giving a sharp interface between dispersion and liquid. An air bubble was then introduced into the solution in the capillary tube C. When a potential of 400 volts was applied between the two electrodes (copper disks) the suspension moved towards the positive electrode, while the bubble moved so as to indicate flow of liquid from the positive to the negative electrode. Thus the liquid phase in the system

* In Fig 4 the sediment volume is plotted against time; sediment volume + settled volume = 25 ml for all suspension samples.

flowed in the opposite direction to the electrophoretic migration of Micronex particles, thereby demonstrating the electro-osmosis of the liquid phase.

If the tube was entirely filled with dispersion, then the direction of movement of the air bubble was the same as the electrophoretic migration

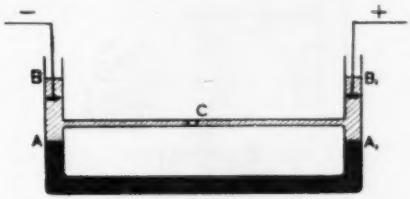

FIG 5

of the carbon particles. Similar results were found for Micronex suspensions in dibromolinoleic acid, oleic acid, and nitrobenzene. In all cases the electro-osmotic flow of the liquid occurred in the opposite direction to the electrophoresis of carbon particles.

Sedimentation Potential in Micronex Dispersions

The existence of a sedimentation potential in a dispersion settling under gravity was demonstrated in a sedimentation cell of the type shown in Fig 6. The cell had 30 ml total capacity, with the same internal diameter as the 25-ml settling vessels used for previous work.[1] Two platinum-wire electrodes were inserted in the tube 2·7 cm apart and at heights corresponding to 9 ml and 15 ml. Except for two narrow vertical observa-

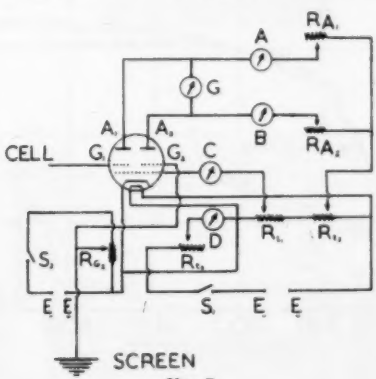

FIG 7

tion strips, the outer wall of the cell was coated with a layer of "Aquadag" and was connected to earth through a brass connexion for electrostatic screening purposes. It was possible to determine the rate of settling and the sedimentation potential of a suspension simultaneously in this cell.

The measurement of an e.m.f. set up in such a cell presents difficult practical problems on account of the very high internal resistance of the

system (10^{10} to 10^{16} ohms). The circuit adopted used a double-tetrode electrometer valve (Ferranti DBM 8A), and is shown in Fig 7. A 12-volt accumulator maintained in good condition was used to supply the heater current through a ammeter D, resistances R_{f1}, and R_{f2}, and a variable

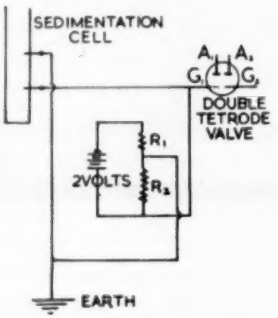

FIG 9

resistance R_{f3}. The latter resistance served to adjust the heater current to its correct value of 0·240 amps. Variable grid bias is provided by the potentiometer R_{g2} across a 4-volt battery, and is supplied between the negative end of the heater and the earthed grid G_2. The e.m.f. to be measured is applied between control grid G_1 and the earth. The variable impedance between the heater and A_1, the fixed impedance between the

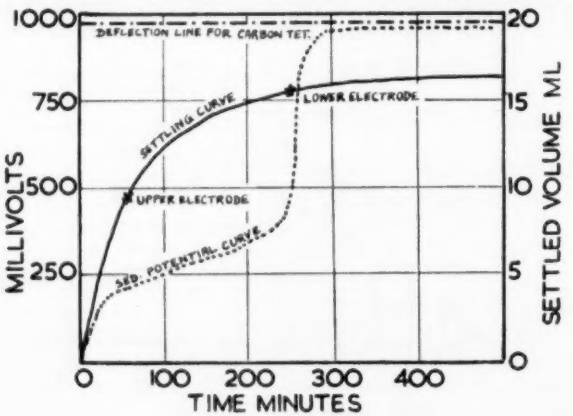

FIG 10 (*a*)

heater and A_2, and the two anode loads form a bridge network. A Cambridge light-spot galvanometer (sensitivity 175 mm/micro-amps and internal resistance of 567 ohms) with a nine-range Universal shunt indicated the out-of-balance voltage of the bridge, and so was a measure of the e.m.f. applied to G_1. The whole unit, Fig 8, was built into an iron box to provide adequate screening.

FIG 6

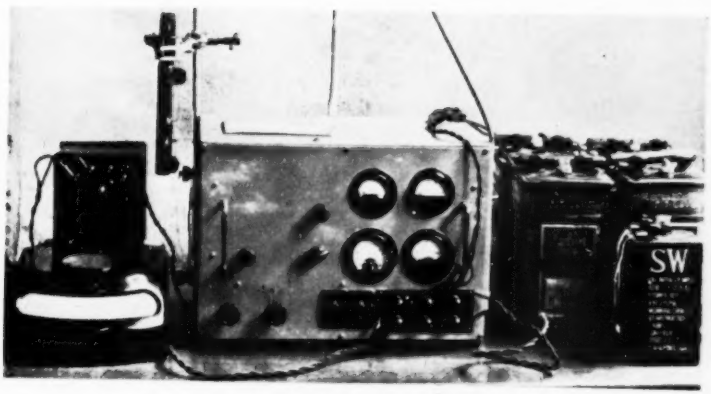

FIG 8

To face p. 994.

Owing to the non-linear characteristics of the valve and the bridge network, direct calibration of the system by application of a known voltage to G_1 was necessary. This was done using the calibration circuit shown in Fig 9. Balance of the bridge and achievement of optimum

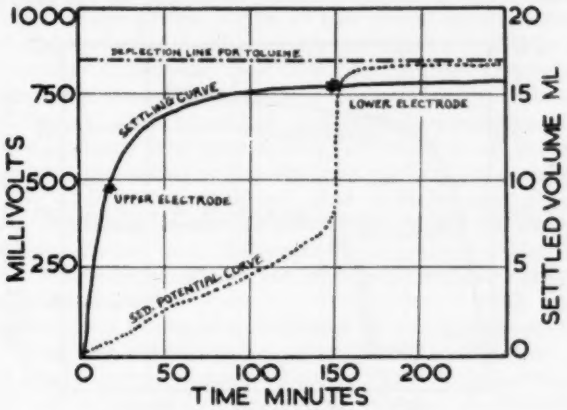

FIG 10 (*b*)

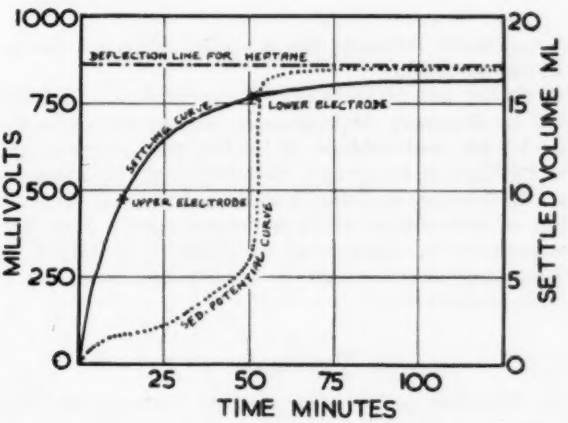

FIG 10 (*c*)

sensitivity could be accomplished by adjustment of the anode load resistances R_{A1} and R_{A2} and the space-charge grid voltage controlled by R_{f1}. Connecting the sedimentation cell, containing the liquid, in the system normally produced an out-of-balance voltage in the bridge due to the voltage drop across the cell caused by the flow of grid current through the liquid. This could be observed from the galvanometer deflection and provided an indication of the electrical conductivity of the liquid. Replacement of the pure liquid by a dispersion now gave rise to a new

galvanometer reading, which, with the height of the suspension interface, was recorded as a function of time as the settling of the suspension proceeded. When the sedimentation was complete, the galvanometer reading always returned to the value corresponding to that for the pure liquid. The difference between the galvanometer deflection for the pure liquid and

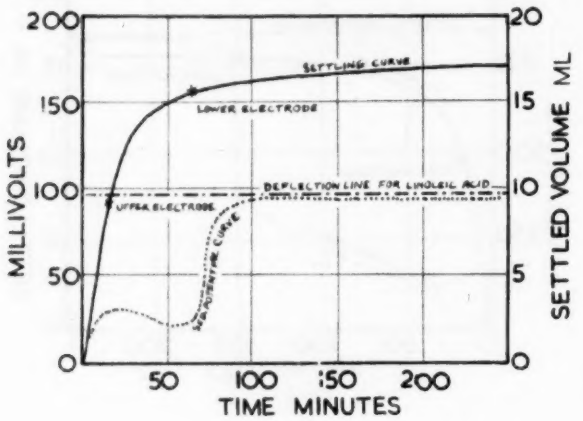

FIG 10 (*d*)

that for the suspension indicated the potential difference due to the sedimentation of the particles.

Figs 10 (*a*), (*b*), (*c*), and (*d*) show the cell potential as a function of time * for a number of Micronex dispersions in carbon tetrachloride, toluene, heptane, and 1·0 per cent solution of linoleic acid, respectively. All the samples used for these measurements were prepared in the usual manner.[1, 2] but the time of ultrasonic agitation was limited to 1 min in order that the sedimentation of the sample could be completed in 2 to 3 hr. Well-dispersed suspensions in commercial additives C, D, and E showed no sedimentation potential, as would be anticipated from the absence of settling in such systems.

DISCUSSION

The results demonstrate the close analogy between the electrokinetic phenomena of carbon-black suspensions in organic liquid media and the colloidal suspensions in aqueous systems. The existence of large sedimentation potentials in dispersions settling under gravity and the effect of externally applied electric fields on the rate of settling and the final sediment volume of the dispersions confirm the view put forward in a previous paper [1] that electrokinetic phenomena play an important role in the stability and the settling behaviour of carbon suspensions.

The electrokinetic phenomena are most marked in liquids of the greatest

* In these Figs the points labelled "upper electrode" and "lower electrode" indicate the times at which the interface passed the corresponding electrodes.

detergent efficiency. Thus the electrophoretic mobility is small in suspensions in toluene or solutions of oleic acid, and large in suspensions in solutions of commercial additives or nitrobenzene, and solutions of dibromolinoleic acid are intermediate. Measurements of electrophoretic mobility thus provide a method of estimating the efficiency of detergent additives.

The origin of the electric forces at the carbon–liquid interface remains a subject of speculation until more experimental evidence is forthcoming. The diffuse ionic atmosphere postulated for aqueous dispersions by Gouy is not acceptable for organosols. The extremely high electrical resistance of organic liquids tends to exclude sufficient concentration of free ions to provide a significant ionic atmosphere, although small concentrations of ions have been postulated to explain such phenomena as dielectric breakdown.[15] It is, however, significant that in most cases the pure liquids of highest dispersing power are also those which are most polar (*e.g.*, nitrobenzene, amino, and halogenated aromatics). For solutions of additives it is also apparent that the more polar additives are more efficient than non-polar molecules.

It has also been shown that mixtures of two strong detergent liquids may be much less efficient than either liquid, should they give rise to electrical double layers of opposite signs. This is well illustrated by the instability and very small final sediment volumes of suspension mixtures of commercial additive E and nitrobenzene. Similar antagonistic effects of two peptizers on the suspendibility of carbon in mineral oils have been noted by Van der Minne.[16]

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THE OCCURRENCE OF MIXED AROMATIC–NAPHTHENIC HYDROCARBONS IN PETROLEUM AS RELATED TO STRUCTURAL GROUP ANALYSIS OF HEAVIER FRACTIONS

By K. VAN NES,* H. I. WATERMAN † (Hon. Fellow), H. A. VAN WESTEN,* and H. VAN KRANEN *

SUMMARY

At present only scattered data are available concerning the occurrence of mixed aromatic–naphthenic hydrocarbons in heavier petroleum fractions, and about the physical and chemical properties of this class of compounds.

To show the importance of this mixed type of hydrocarbons three narrow-boiling gas-oil fractions of widely divergent types were investigated. The aromatic portions were isolated chromatographically and then analysed by the Direct Method (elementary analysis and molecular-weight determination before and after hydrogenation). The figures obtained show that the greater part of the aromatic molecules of petroleum contain naphthene rings. Therefore the reliability of the analysis of petroleum products by methods which neglect these mixed-type compounds is questionable.

DURING the last twenty years there have been published various methods for analysing petroleum fractions, which are based on data about synthesized hydrocarbons. The model substances taken as a basis for such methods must be true representatives of the components of the oils to be analysed. Since in the gasoline range a fairly good insight has been gained into the composition, and the properties of hydrocarbons in this range are relatively well-known, it might be expected that a reliable group of representatives can be found for gasolines. In the heavier fractions, however, the present situation is such that, broadly speaking, only those hydrocarbons which are relatively easy to synthesize are studied. Unfortunately, such types of hydrocarbons are in general not the more abundant components in the heavier petroleum fractions.

In this connexion the authors would like to call attention to a class of hydrocarbons usually referred to as mixed aromatic–naphthenic compounds, *i.e.*, hydrocarbons containing aromatic rings, naphthene rings, and alkyl chains side by side. In the authors' opinion there is convincing evidence in the literature that this class of hydrocarbons is quite well represented in the heavier petroleum fractions.[1, 2] From a synthetic point of view, however, it has received only limited attention. Perhaps this explains why several new methods for the analysis of mineral oils have been proposed in the literature which give reliable results only if aromatic components containing naphthene rings occur in negligible amounts in crudes.

An example is the M-n_D^{20} method developed by Hersh, Fenske, *et al* [3] for the analysis of oil fractions. As indicated by the name of the method, only the measurement of molecular weight (M) and the refractive index

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(n_D^{20}) is required. Formulæ derived from molecular weights and refractive indices of synthesized hydrocarbons are used for the analysis.

As formulæ are given only for naphthene/paraffin mixtures and for aromatic/paraffin mixtures, the oils to be analysed have to be separated beforehand into aromatic and paraffin/naphthene portions, *e.g.*, by chromatography.* Although Hersh, Fenske, *et al*, realized that mixed-type compounds may occur in petroleum fractions, the procedure of the M-n_D^{20} method is based on the assumption that these hydrocarbons are scanty. The authors doubt whether this assumption is justified.

In order to give further support to the view that aromatic compounds in petroleum actually contain for the greater part naphthene rings, the aromatic portions of various oils of widely divergent type were investigated by the Direct Method,[2] *i.e.*, by measuring elemental composition and molecular weight before and after hydrogenation. Although the results obtained by the Direct Method are not in all details indisputable, the authors are convinced that it is to date the most reliable method for the analysis of petroleum fractions beyond the gasoline range.

As an illustration of the results obtained the investigation will be discussed of three rather narrow-boiling gas-oil fractions of a paraffinic Indonesian crude (Borneo), a Pennsylvanian crude, and a highly cyclic Indonesian crude (Tarakan), respectively. In Table I a few details are given on these three gas-oil fractions. In this table the subscripts A, N, and P refer to aromatics, naphthenes, and paraffins, respectively, per cent C_A indicates the number of carbon atoms in aromatic-ring structure per 100 carbon atoms in the sample; R_A is the number of aromatic rings per mean molecule of the sample and R_T the total number of rings per mean molecule: $R_T = R_A + R_N$.

TABLE I

Physical Properties and Structural Group Analysis of the Three Original Oil Fractions

	Borneo	Pennsylvania	Tarakan
Physical properties :—			
Boiling range (in ° C at 760 mm)	*ca* 330–340	*ca* 305–340	*ca* 315–340
Refractive index n_D^{20}	1·4666	1·4637	1·5212
Density d^{20}	0·8299	0·8319	0·9360
Molecular weight M	285	265	248
Carbon distribution (Direct Method) :—			
% C_A	12·8 *	7·8	28·1
% C_N	7·4 *	22·6	42·1
% C_P	79·8 *	69·6	29·8
Ring content (Direct Method) :—			
R_A	0·44 *	0·25	0·85
R_N	0·28 *	0·71	1·81
R_T	0·72 *	0·96	2·67

* Analysis by n-d-M method.[3]

The figures for carbon distribution given in Table I show that the ratio per cent C_N/per cent C_A varies from 0·58 (Borneo) to 2·90 (Pennsylvania)

* In this respect the M-n_D^{20} method is quite analogous to the methods of Lipkin, Martin, and Kurtz.[4]

and the paraffinic carbon content from 80 per cent (Borneo) to 30 per cent (Tarakan). It may be assumed therefore that these three samples are reliable representatives for all possible gas oils in this boiling range.

By percolation through silica gel the three oils were split up into aromatic and non-aromatic (saturated) portions,* applying pentane development and alcohol displacement. The saturated portions were not further investigated.

After stripping off the alcohol—by heating on the steam-bath at 5 mm Hg in a nitrogen stream—the aromatic portions were hydrogenated in pentane solution with Ni on guhr as the catalyst. First a hydrogenation at 200° C and a pressure of *ca* 250 atmospheres was carried out. After replacement of the catalyst by a fresh sample, hydrogenation was repeated at 250° to 275° C and a pressure of 220 to 250 atmospheres. The last traces of spectroscopically detectable aromatics were removed from the hydrogenated oils by percolation through silica gel. Ultra-violet spectra of the oils showed them to be wholly free of aromatics.

Both the original aromatic portions and the hydrogenated oils were then analysed for elemental composition, molecular weight, refractive index, and density. The oxygen determinations were carried out according to the Schütze–Unterzaucher method.[5] The molecular weights were determined ebullioscopically.[6] A survey of these data is represented in Table II.

TABLE II

Elemental Analysis and Physical Properties of Aromatic Portions before and after Hydrogenation

Oil fraction	% H	% C	% O	% S	% total	n_D^{20}	d^{20}	M
Before hydrogenation :—								
Borneo	9·05	89·19	1·4	0·43	100·07	1·5837	1·0086	244
Pennsylvania	10·25	88·09	1·6	0·33	100·27	1·5436	0·9595	242
Tarakan	9·55	89·14	0·8	0·26	99·75	1·5632	0·9938	242
After hydrogenation :—								
Borneo	13·06	86·77	—	—	99·83	1·4886	0·9047	243
Pennsylvania	13·36	86·69	—	—	100·05	1·4814	0·8869	248
Tarakan	12·94	87·00	—	—	99·94	1·4904	0·9113	245

For the oils mentioned in Table II the carbon distribution and ring content were calculated by the Direct Method [3] and by the M-n_D^{20} method.[2] The following formulæ were used :—

Direct Method—

$$\% C_A = [1201(M'H' - MH) - 201{\cdot}6(M'C' - MC)]/1{\cdot}008MC$$

$$R_A = 0{\cdot}002479(M'H' - MH) - 0{\cdot}0004163(M'C' - MC) - 0{\cdot}5 \quad \text{(valid for } R_A \geqslant 1\text{)}$$

$$R_T = 1 + 0{\cdot}00579M'(14{\cdot}37 - \mathrm{H}')$$

$$\% C_R = 240200(2\,R_T + \mathrm{R}_S)/M'C'$$

($R_S = R_T$ for $R_T \leqslant 1$ and $R_S = 1$ for $R_T \geqslant 1$)

* Various chromatographic procedures may be suitable for this separation; the method described by Hersh, Fenske, *et al* was closely followed.

In these formulæ H and C represent the percentages of hydrogen and carbon respectively, while the dash denotes the hydrogenated state.

M-n_D^{20} method—

Aromatic oils

$$\%\ C_A = \frac{2150[(n_D^{20} - 1{\cdot}4750)M + 8{\cdot}79]^{0{\cdot}56}}{M - 3 + 2{\cdot}3[(n_D^{20} - 1{\cdot}4750)M + 8{\cdot}79]^{0{\cdot}56}}$$

$$R_A = 0{\cdot}165\ [(n_D^{20} - 1{\cdot}4750)M + 8{\cdot}79]^{0{\cdot}74}$$

These formulæ hold good for compounds with kata condensed * six-membered rings.

Hydrogenated oils

$$\%\ C_R = \frac{2920[(n_D^{20} - 1{\cdot}4750)M + 8{\cdot}79]^{0{\cdot}73}}{M - 2 + 0{\cdot}57[(n_D^{20} - 1{\cdot}4750)M + 8{\cdot}79]^{0{\cdot}86}}$$

$$R_T = 0{\cdot}284\ [(n_D^{20} - 1{\cdot}4750)M + 8{\cdot}79]^{0{\cdot}86}$$

These formulæ hold good for mono-, di-, and tri-cyclic naphthenes, with one paraffinic side-chain per molecule and are averaged for condensed and non-condensed rings.

A survey of the results of these calculations is given in Table III.

TABLE III

Structural Group Analysis of the Aromatic Portions of Three Gas Oils before and after Hydrogenation

	Direct method		M-n_D^{20} method		Difference between M-n_D^{20} and direct method	
	% C_A	R_A	% C_A	R_A	Δ % C_A	Δ R_A
Before hydrogenation :—						
Borneo	59·1	2·18	61·3	2·30	+2·2	+0·12
Pennsylvania	44·8	1·49	52·0	1·81	+7·2	+0·32
Tarakan	49·8	1·74	56·8	2·05	+7·0	+0·31
	% C_R	R_T	% C_R	R_T	Δ % C_R	Δ R_T
After hydrogenation :—						
Borneo	76·1	2·84	73·3	2·42	−2·8	−0·42
Pennsylvania	65·9	2·45	64·4	2·12	−1·5	−0·33
Tarakan	79·6	3·03	74·7	2·50	−4·9	−0·53

It is striking that the per cent C_A and R_A figures for the aromatic portions of the gas oils found by the M-n_D^{20} method are all higher than those found by the Direct Method and that the per cent C_R and R_T figures for the hydrogenated oils are all lower than those found by the Direct Method.

If no naphthene rings were present in the aromatic molecules, the per cent C_A for the oils before hydrogenation should be equal to the per cent C_R after hydrogenation. Both methods give rather large differences for these

* Kata condensation means that there is one common bond in two condensed rings, as in the series : naphthalene, anthracene, tetracene, etc.

figures, which indicates that naphthene rings occur in reasonable amounts in the aromatics.

Comparison of the per cent *C* and *R* figures found by the Direct Method before and after hydrogenation shows that the Borneo, Pennsylvanian, and Tarakan aromatics contain on an average 0·66, 0·96, and 1·29 naphthene rings per molecule; in other words, that of the total number of rings 23, 39, and 43 per cent, respectively, are naphthene rings.

It may be concluded that it is not justified to assume that naphthene rings are present only in negligible amounts in aromatics. For the development of reliable methods for analysing aromatic portions of the heavier oils based on data of synthesized hydrocarbons a further study of mixed aromatic–naphthene hydrocarbons is required.

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Amsterdam, February 1952

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Oilfield Exploration and Exploitation

Geology

2988. Production and development in the United States during 1951. Anon. *J. Petrol. Tech.*, Mar. 1952, **4** (3), 11–12.—In 1951 U.S. crude production was about 2,228,000,000 brl, 13% more than in 1950. In addition 241,000,000 brl of natural gas liquids were produced. Over 8,000,000,000 M.c.f. of gas was produced, and over 50% was transported to industrial and domestic centres. North Dakota began to produce oil in 1951. 57% of the liquid hydrocarbon production came from the Gulf Coast of Texas and Louisiana.

Nearly 45,000 wells were drilled in 1951, about 1400 being service wells. There were over 23,000 oil wells and 3100 gas wells. More than 10,000 wildcats were drilled, nearly 1000 finding oil and over 200 gas. 600 oil wells and 100 gas wells proved extensions or new horizons. On 1 Jan. 1951 there were nearly 450,000 oil wells. New finds, revisions, and extensions added over 1,000,000,000 brl of oil to reserves.

The oil discovery in the Williston Basin of North Dakota was in the Devonian and Mississippian. There are also possibilities in the Ordovician, Silurian, Jurassic, and Cretaceous. There may be anticlinal and stratigraphic traps. There are also reef developments. Twelve oilfields and two gas fields were found in Colorado, and three oilfields in Nebraska. 1000 rigs operated in West Texas, 250 being on the Spraberry trend, which now has seventeen areas of production, but many of these may coalesce on further work. Discoveries were made along a trend just east of the Central Basin Platform in the Lower Permian, Pennsylvanian, and Ordovician. Twenty-five new oil and gas fields were found in the Anadarko Basin. Wells were drilled and abandoned in Nevada, Arizona, and Oregon, while a small oil well was completed in Washington. A relatively shallow discovery was made in southern Alabama. In California 577 exploratory wells yielded seven new oilfields, two gas fields, and eleven extensions or new reservoirs. G. D. H.

2989. Significance of oil and gas seeps in world oil exploration. W. K. Link. *Bull. Amer. Ass. Petrol. Geol.*, 1952, **36**, 1505–40.—Many great oilfields are the result of seepage drilling, and examination shows that oil and gas seepages gave the first clues to most of the oil-producing regions. Seepages are most numerous in the youngest sediments, especially where they have been folded, faulted, and eroded, and on basin margins, and many are the result of the destruction of major accumulations of oil reservoirs. Geologists can see exposed a great many "type oil accumulations" by studying seepages and the reasons for their location: oil and gas seepages fill one of the prerequisites for an oil-producing area, *i.e.*, source rocks, and since large seepages usually result from pool destruction, they also indicate reservoir rocks and structure.

The recent developments in Western Canada, the Uinta Basin of Utah, and the Cuyama Valley in California are examples of the value of seepages in geological exploration. E. N. T.

2990. Geology and oilfields of Brazil. E. F. Taylor. *Bull. Amer. Ass. Petrol. Geol.*, 1952, **36** (8), 1613–26.—More than a million sq. miles of Brazil's 3½ million sq. miles area is occupied by large sedimentary basins, ten of which have petroleum possibilities. Geological, geophysical, and drilling operations are under way in these sedimentary areas under the direction of the National Petroleum Council of Brazil. Nine oilfields have been discovered in Brazil, in Salvador, Bahia, and daily production in Bahia is 2500 brl, with equal refining capacity, and present trends in Brazil are towards an expanded, accelerated development programme by the Brazilian Government, under the direction of the National Petroleum Council. E. N. T.

2991. Sedimentology and petroleum geology. D. J. Doeglas. *Proc. Third World Petrol. Congr.*, 1951, **1**, 439–45.—The advantages and limitations of certain investigations concerning sedimentology are discussed. Reference is made to grain-size variation and distribution, the shape and rounding of grains, mineral association, and thin sections.

For a complete understanding of the geology of sedimentary formations, further research is necessary regarding the distribution of mineral associations in basins where

good stratigraphic zones occur. It is also remarked that more knowledge of sediments and sedimentation is badly needed in order to deal with problems on petroleum geology, and this knowledge can be gained only by large-scale studies. G. S. S.

2992. Facies shift and isochronous correlation. J. Dufour. *Proc. Third World Petrol. Congr.*, 1951, **1**, 428–38.—The palæontology of recent sediments has proved of much importance in the interpretation of the spatial distribution of older formations and in elucidating the geological history of sedimentary basins or embayments.

In assessing the possibilities of oil accumulation, the distribution of facies plays an important part. A correct understanding of the depositional history is therefore essential, and this includes an analysis of the various types of sediments in relation to their environment.

Bio-stratigraphic studies have been useful in interpreting both vertical and lateral variations of the various facies-units or types of facies distinguished. The outcome of such studies is of primary importance for regional correlation of sedimentary units deposited during a palæontologically defined time interval. The isochronous correlation thus obtained may lead to the construction of palæo-geographical maps which might be of much assistance in the search for new oil provinces. An interesting case was revealed in Northwest Venezuela by electrical (Schlumberger) logging in the Palæocene, whereby it was possible, with a reasonable degree of certainty, to establish isochronous correlation. G. S. S.

2993. Bacteriology and petroleum. J. Appert. *Rev. Inst. franç. Pétrole*, June 1952, **7** (6), 170–80.—Living bacteria have been found in most sediments examined, irrespective of the depth of the water or distance from the land. Their numbers diminish with depth in the sediments, but they were still present at a depth of 7 m. However, they are dependent on the nature of the sediments, and may increase with a change in the sediments. Ærobic bacteria have been found where there is no free oxygen; presumably they are in a resting state. At depths of more than 15 cm anærobes are usually predominant. Activities are reduced at low temp, under low water contents, and when the organic matter is reduced in amount.

Bacteria have been found in rocks at depths of 300 to 2100 m in rocks of various ages in petroliferous strata at Saratou and Bougourouslan, but they were rare in non-petroliferous strata. They are claimed to have been found at 475 m in a limestone–anhydrite–sulphur series under conditions said to exclude contamination. It is not possible to demonstrate that bacteria found at depth are the direct descendants of those entombed with the sediments. Sulphate reducers, and methane and hydrogen producers have been observed. At normal pressures 85° C may be the limit for living bacteria; high pressures may modify this.

Methane is a common bacterial product, and the optimum temp is 30° to 45° C. There are suggestions of the formation of small amounts of ethane and propane under pressures of 200 to 500 atm. Higher hydrocarbons have been produced from fatty acids. Some bacteria form pigments containing hydrocarbon complexes. Zobell notes that the greater part of the non-gaseous hydrocarbons is associated with bacterial protoplasm.

Some bacteria can dislodge hydrocarbons from solid surfaces mechanically or by means of surface tension reduction and gas formation. So far the action has been observed only in the laboratory. Bacteria can destroy hydrocarbons. Ærobes are particularly active, and sulphate-reducers are also effective. The products include CO_2, bacterial protoplasm, pigments, acids, alcohols, etc. Paraffins may be more easily attacked than cyclic compounds.

Micro-biological prospecting for oil has been attempted. Fluorescence and dyes have been used to identify bacteria which attack certain hydrocarbons. Alternatively, bacterial products may be sought in this form of oil prospecting.

Proposals have been made for analysing mixtures of hydrocarbons by the use of specific bacteria, and for using bacteria to form acids from gaseous hydrocarbons in the presence of traces of heavy metals. Bacteria may adversely affect petroleum products. G. D. H.

2994. Contributions of bacteria to the origin of oil. C. E. Zobell. *Proc. Third World Petrol. Congr.*, 1951, **1**, 414–20.—Biochemically and ecologically, versatile bacteria, by their ability to catalyse so many organic and inorganic reactions, are well adapted to

affect recent marine sediments. Evidence suggests that bacteria may also be active in older sediments containing oil, water, and minerals essential for the growth of these micro-organisms.

Assuming that oil has originated from organic remains, it is almost axiomatic that bacteria have contributed to its formation, because all types of organic matter are susceptible to bacterial modification. Bacteria, under anærobic conditions, tend to result in residues relatively richer in carbon or hydrogen and consequently more petroleum-like in chemical composition. Bacteria may also contribute to the origin of oil by catalysing the hydrogenation of certain compounds.

Several mechanisms are referred to by which bacteria may advance the liberation of oil from oil-bearing materials and thus promote its migration and accumulation.

G. S. S.

2995. On conditions favouring the preservation of chlorophyll in marine sediments. M. Brongeresma-Sanders. Appendix W. G. Aldershoff. *Proc. Third World Petrol. Congr.*, 1951, **1**, 401–13.—The hypothesis is put forward that hypertrophic conditions in the sea are probably the main cause for the origin of oil, and are of much greater importance than persistent stagnation. In eutrophic waters the production of both plankton and benthos are great, and a direct relation exists between them. Above a certain max, the relation is indirect; the greater the plankton production the scarcer the benthos. Such conditions are termed hypertrophic. Their distinctive features are a frequent occurrence of thick waterbloom (particularly of red water) and of mass extermination of invertebrates and vertebrates.

Hypertrophic conditions may be produced by (*a*) intense upwelling, occurring in the open sea and in some inland seas, as the Caspian and Red Sea; (*b*) accumulation of nutrients derived from the land: this occurs only in semi-separated parts of the sea.

In both instances conditions will have been more intense and more frequent in one geological epoch than in another. Consequently, hypertrophy in one period is more important than in others, though it must be emphasized that the occurrence of hypertrophy does not require a combination of factors that is only rarely attained. Persistent stagnation only occurs in certain basins, and consequently is rare.

G. S. S.

2996. Radioactivity and the origin of petroleum. I. A. Breger and W. L. Whitehead. *Proc. Third World Petrol. Congr.*, 1951, **1**, 421–7.—In 1942 the American Petroleum Institute initiated research at the Massachusetts Institute of Technology to study the effects of radioactivity on components of marine organic deposits.

This paper summarizes a number of important results so far obtained. (1) Bombardment of fatty acids with α particles leads to the production of aliphatic hydrocarbons contained in petroleum; (2) bombardment of a naphthenic acid leads to the formation of a naphthenic hydrocarbon which is found in crude oil; (3) unsaturated compounds which are bombarded may be hydrogenated during the course of the reactions; (4) bombardment of almost any organic compound results in the formation of gaseous hydrocarbons found in association with crude oils.

Progress has also been made on (*a*) the determination of the association of radioactive elements and organic matter in marine organic shales, and (*b*) detailed measurements of the radioactivity of a number of important formations associated with petroleum.

G. S. S.

2997. Stratigraphical correlations with microfacies in western Aquitain. J. Cuvillier. *Proc. Third World Petrol. Congr.*, 1951, **1**, 446–8 (in French).—Petroleum prospecting by the Société Nationale des Pétroles d'Aquitaine has been carried out by means of a system of stratigraphic correlation in which microfacies proved much more important than microfauna. Numerous thin sections have enabled detailed stratigraphic subdivisions to be made embodying all the most characteristic microfacies. By the aid of many photographs, mainly Jurassic, Cretaceous and the Nummulitic, all the chief micro-petrographic aspects of the Secondary–Tertiary sequence are illustrated.

The microfacies thus presented will possess a more than regional and Aquitanian value; in fact, a number of them can be observed throughout the whole Tethyan area of sedimentation.

Paper has been published in full as supplement of the *Proceedings* of the Congress. It contains 108 pp. and ninety plates. From the publishers, E. J. Brill, Leiden, Netherlands. G. S. S.

2998. Method of study and interpretation in a predominantly calcareous sequence. A. Lombard, M. Gottis, Y. Jullian, and M. Mainguy. *Proc. Third World Petrol. Congr.*, 1951, **1**, 449–62 (in French).—A recently developed method of correlation between surface and/or subsurface logs has been tested mainly in a predominantly calcareous sequence. The markers are integrated with serial observations made from detailed and continuous analysis of surface sections and subsurface logs. From this a clear picture is obtained of the lithologic succession. Such a picture is termed a " sequence " and its detailed study a " sequence analysis " ; it provides a useful tool which can be adapted for both surface and subsurface sections, and for their comparison. Examples are given of tests carried out on a series of dominantly calcareous rocks in Languedoc Mediterranean. The paper concludes with a note on its practical applications with a view to the genetic definition of facies, thereby contributing to palæogeography. G. S. S.

2999. Correlation, age determination, and the Tertiary pelagic foraminifera. T. F. Grimsdale. *Proc. Third World Petrol. Congr.*, 1951, **1**, 463–75.—A three-year study has been made of foraminifera from the Tertiary rocks of the Middle East, and endeavours have been made to base stratigraphic interpretations mainly upon the sequence of pelagic species. These have previously provided the most reliable means of age determination in the Tertiary sediments of the western hemisphere, and the extension of this method to the Middle East is the principal subject discussed. A chart is given in an attempt to compare the stratigraphic distribution of a number of species of Tertiary pelagic foraminifera in the two provinces : Gulf of Mexico–Caribbean Sea and the Middle East.

Important research is also proceeding regarding the study of the pelagic foraminiferal sequence in the West European Tertiary deposits. A definite correlation of the American and European Tertiary by means of the pelagic foraminifera would be of much service to workers who attempt to achieve correlation over long distances. G. S. S.

3000. On some Paleocene and Eocene larger foraminifera of Western Venezuela. B. van. Raadshooven. *Proc. Third World Petrol. Congr.*, 1951, **1**, 476–89.—A study has been made of the more typical assemblages of larger foraminifera found in the Paleocene and Eocene of Western Venezuela. Discussion is given to the generic position of the Paleocene species " Lepidocyclina " barbadensis Vaughan, and to the nepionic development of closely related species of Lepidocyclina from two different lower Middle Eocene faunas.

The author remarks that he has taken into account only certain of the more important general results which are closely related to recent work on the larger foraminifera from the Caribbean region. Though no new names are now proposed for the new species, it is hoped to publish shortly a more complete discussion of the assemblages studied, including descriptions of the new species. G. S. S.

3001. Microfauna of the Upper Cretaceous–Paleocene—their application in French Equatorial Africa. M. Lys and N. Grekoff. *Proc. Third World Petrol. Congr.*, 1951, **1**, 490–6 (in French).—Report on the application of a comparative study of the foraminifera and ostracoda to the subdivision of the Upper Cretaceous–Paleocene, giving special attention to a little explored region of the African continent. Attempts are also made to elucidate the evolution of this microfauna in the Cameroon Basin in connexion with its migrations in the Atlantic and Mediterranean provinces. These migrations were responsible for the progressive appearance in time of the genera and species in the various basins of that province. Accompanying the paper is a stratigraphic table of the microfauna in the Cameroon Basin. G. S. S.

3002. Structure and origin of Northern Sangre de Cristo Range, Colorado. J. W. Gabelman. *Bull. Amer. Ass. Petrol. Geol.*, 1952, **36** (8), 1574–612.—An imbricate zone of east-dipping thrusts associated with parallel, normal, and transverse faults

on both sides of the range was disclosed in the Lower Paleozoic sediments diagonally crossing the northern Sangre de Cristo Range, Colorado. The steep western slope of the range is considered the scarp of a normal fault, and two areas offer evidence for shallow thrusting confined to the Lower Paleozoic: transverse faults commonly displace thrusts. Open folds sharpen southeastward from Wellsville and break into thrusts, and the folds at Orient also break into thrusts to the north and appear to denote the southern end of the thrust zone.

The zone of westward thrusting is part of a broader zone, including the Pleasant Valley fault and similar thrusts west of the San Luis Valley, and is probably continuous with the early Eocene London–Weston thrust belt of South Park and the Mosquito Range. The northeast thrusts have no counterpart in the northern range, but large normal faults in the Wellsville–Orient thrust zone indicate vertical uplift of a pre-Cambrian area and are correlative with late Eocene uplift on the south. Downfaulting of the San Luis Valley from Oligocene to Recent outlines the range's present trend and explains its divergence from the thrusting zone. E. N. T.

Geophysics and Geochemical Prospecting

3003. Precision of travel-time curves in the seismic method of geophysical surveying. A. A. Fitch and E. H. Lloyd. *Proc. Third World Petrol. Congr.*, 1951, **1**, 497–513.—Attention is drawn to the sources of difference between seismic interpretation and the actual geological structure of the subsurface. In two groups of examples quoted the accuracy of subsurface dips is a little greater than that customarily attained by surface measurements.

The standard error of an estimate of dip made with a particular seismic technique is expressed as a function of a number of quantities, some of which are parameters related to the details of the field technique. Thus it becomes possible to ensure that a specified level of accuracy is attained by suitably arranging the field technique.

It is shown that the variance of the estimate of dip can be used as the basis for grading reflections, and comparisons are made with the Gaby system.

An instrument is described for computing a "precision factor" related to the variance. G. S. S.

3004. Form and laws of propagation of seismic wavelets. N. Ricker. *Proc. Third World Petrol. Congr.*, 1951, **1**, 514–36.—In the past, seismic methods have seen wide and improved application to petroleum exploration, but subsequent attempts to obtain further improvement have met with serious obstacles, some of which have not yielded to the customary experimental procedure of development. Recently, however, an extensive series of experimental studies have been carried out to determine the true form and nature of the seismic disturbance which travels outward from an explosion at a point in the earth.

The material selected was a thick homogeneous shale series: Pierre Shale of Cretaceous age, Denver Basin. The results gave a good understanding of the form and character of the primary seismic disturbance and its laws of propagation. It is concluded that, in shale, agreement of experiment with the wavelet theory is very satisfactory. Numerous figures, including wave surface charts and seismograms, accompany the paper. G. S. S.

3005. Seismic surveys over asymmetrical structures in S.W. Persia. D. T. Germain-Jones. *Proc. Third World Petrol. Congr.*, 1951, **1**, 546–63.—The Asmari limestone anticlines which form the oil reservoirs in Southwest Persia offer an attractive target for geophysical work, and a description of these structures is given. Difficulties met with in their location are considered, together with brief details of the refraction technique developed for this large-scale problem. It has been found that more accurate delineation of these structures is needed and more detailed information required on the behaviour of their flanks. An examination of the possibilities of improving the interpretation technique has followed, and examples of the results obtained over theoretical structures are given.

Marked discrepancies have arisen between reflection and refraction results over the same anticlinal structures. Down to about 4000 ft the results of two surveys

furnish similar pictures, but at greater depths of 12,000 ft or more there is an increasing divergence, the refraction axis being displaced northeast of the reflection axis. An example of this discrepancy is afforded by surveys made over an anticline near the town of Ahwaz. G. S. S.

3006. Surface measurement of gamma radiation in a geochemically mapped area. J. Hubicki and Z. Wierzbicka. *Bull. Main Petrol. Inst.*, **5** (1) (Suppl. to *Nafta*, 1951, **7**), 17–19.—Search for oil in 1950 was conducted taking samples of earth and occluded atmosphere and testing them for hydrocarbons by chemical methods. Their places of origin were later tested for γ-rays. Subterranean hydrocarbons show but little more γ radioactivity than the surrounding earth, but on exposure to cosmic radiation they become distinguishable by means of electronic equipment (G. M. Counters). Samples were taken from 2·5 m below cultivation level from area described by S. Sulimirski *et al* in "Geochemical surface measurements," *Proc. Main Petrol. Inst.* (11). Experiments were conducted in the field. After subtracting background radiation results were plotted, and they correspond fairly well with "bitumen index."
A map, section, and table are given. M. S.

3007. Patents. U.S.P. 2,590,873 (24.10.39; 1.4.52). S. Krasnow and L. F. Curtiss, assrs to Schlumberger Well Surveying Corpn. Method and apparatus for measuring radioactivity.

U.S.P. 2,590,874 (24.8.42; 1.4.52). S. Krasnow and L. F. Curtiss, assrs to Schlumberger Well Surveying Corpn. Multiple element radioactive ray recording.

U.S.P. 2,590,930 (16.2.49; 1.4.42). G. J. Butterworth, assr to Sperry-Sun Well Surveying Co. Circuit control for well surveying instruments.

U.S.P. 2,591,177 (2.12.49; 1.4.52). W. H. Mayne, assr to O. S. Petty. Seismic surveying apparatus having means for detecting improper connexion of a seismometer with the line leading to a remote recorder.

U.S.P. 2,591,192 (29.11.49; 1.4.52). J. O. Parr, assr to O. S. Petty. Seismic surveying apparatus with means for detecting faulty connexions beween a seismometer and the line leading to a remote recorder.

U.S.P. 2,592,125 (18.11.49; 8.4.52). H. G. Doll, assr to Schlumberger Well Surveying Corpn. Well logging apparatus for logging static spontaneous potentials in wells. R. C. R.

Drilling

3008. Dampener cuts mud pump surges 78%. R. L. Walker. *World Oil*, June 1952, **134** (7), 155.—One of the primary causes of mechanical failure is fatigue. Pump surges necessarily affect the major part of a rig assembly from the pump motor, through the mud lines to the mud hose and drill string. Furthermore, pump surges, if they be transmitted to the mud column, are detrimental to the lifting of bit cuttings. The use of a dampener is therefore one way of reducing general wear.

The desurger described may be installed at any of several points on the rig, and operates on the principle of absorbing excess energy by the throttling of the mud through numerous orifices in a mandrel, concentric with which is a rubber sleeve supported by gas pressure. The combined effect of perforations and sleeve have satisfactorily dampened the surges by as much as 78%. Thus the pump may be used at slightly above its initially rated value, or at least the life of the unit may be extended. A. J. H.

3009. Mechanics of cable tool drilling. K. N. Mills. *World Oil*, Sept. 1952, **135** (4), 123.—The principles underlying cable tool drilling are essentially problems of vibrational mechanics. The advances made in technique of drilling have been due primarily to improvements in mechanical design. The mechanism of drilling may be divided into the wt of tool; spring, or drilling line; and secondary spring, being the shock absorber installed at the surface. The natural frequency of such a system may be expressed empirically as

$$f = 188\sqrt{\frac{KS}{W(K+S)}}$$

where K is spring constant for drilling line; S is spring constant for the shock absorber; W is the wt of the tool and one-third wt of drilling line.

The vertical travel of the tool, greater than the travel at the surface, is dependent of the characteristics of the drilling assembly, the friction losses within the hole, and the energy dissipated in drilling the formation. Ideally, the max effect is obtained when drilling near the natural frequency of the system; if, however, this is unsuitable, then the higher the tool velocity, the greater the available energy for drilling.

The relationships between f, W, and depth of drilling for ¾-, ⅞-, and 1-inch lines are given graphically. A. J. H.

3010. Uses and advantages; jet-nozzled rock bits. L. L. Payne and R. H. Nolley. *World Oil*, June 1952, **134** (7), 119.—The idea of using jet bits for drilling soft formations has existed for some time, but the combination jet-nozzled bit with rollers had not met with much success until recently (1947). It is generally agreed that the failure was due to the insufficiently high jet velocity. In the conventional rock bit the jet is used for the purpose of cooling and cleaning the rollers, but in the above type the jet is directed at the bottom of the hole, the turbulence thereby created being a means of cooling and cleaning the bit-rollers.

Since these bits require jet velocities in the region of 200 ft/sec (as opposed to a max of 100 ft/sec in conventional bits), the selection of adequate pumping units becomes of greater importance. The pressure drop in a system may be estimated, and from the max capacity of a pump, the max operating depth to which to use jet bits deduced. In some cases pump liners are changed for different drilling depths, or the pumps may be assembled in parallel or in series for the greater delivery pressure and flow. Jet bits are not suited to hard formations, and the added expense is seldom justified.

Data are given for the estimation of max drilling depth using certain jet bits. From these data are calculated specimen examples; in general, a larger pipe gives a positive hydraulic advantage for this type of drilling. A. J. H.

3011. Calculating economical hook horsepower. J. D. Goodrich. *World Oil*, Aug. 1952, **135** (3), 141.—Rule of thumb methods are much used for the estimation of power requirements: providing 100 h.p/1000 ft, or ensuring sufficient power to be able to hoist at the rate of 100 f.p.m. off bottom. Both assumptions have their disadvantages. The hoisting speed is dependent actually on two conditions; when coming out of hole from bottom, the speed is a function of the wt of the tools, and beyond a certain point the max speed is also the max speed of the rig. Expressing this in an equation, and integrating, permits the estimation of required time of pulling out. This method has more to recommend it than the previous methods. A. J. H.

3012. Giant magnetic fishing tool has wide application. G. W. J. Trowbridge. *World Oil*, Apr. 1952, **134** (5), 152.—Small junk left in wells have given rise to much trouble in recovery. The original suggestion of an electromagnet was discarded because of practical details, but recently, the use of a suitable permanent magnetic material has been extended to general fishing problems. The use of the magnets, which have a pull of approx 45 p.s.i., have been demonstrated in several fields; examples are cited. In general, the strength of the magnet outlives the mechanical structure of the tool. Loss of magnetization is small; a keeper ring is needed when the tool is not in operation. A. J. H.

3013. Automatic drilling comes of age. M. E. True. *World Oil*, Sept. 1952, **135** (4), 131.—A general review of the development of automatic tongs, spinners, stabbers, rackers, elevators, and other rig equipment. The advantages of such units are obvious in preventing fatigue of crew when operating deep well rigs. A. J. H.

3014. Location of oil prospects by use of the drill. W. M. Marshall. *J. Petrol. Tech.*, Mar. 1952, **4** (3), 17.—Analysis of the means of reducing drilling costs shows that they fall into three groups: (1) equipment; (2) techniques; (3) rate of penetration. A 150-h.p. portable outfit with 90-ft derrick using 3½-inch externally upset "J" grade drill tubing weighed only 70,000 lb and drilled eleven 6¾-inch holes to depths of 1800 to 5600 ft. This caused a saving of about $5/ft. Two larger portable rigs are used for deeper and larger holes (8800 ft with 4½-inch tubes). The mud pumps might

be reduced in wt and size. A wire-line, side-wall sampler has been planned and also a side-wall sampler for reservoir fluids. Diamond coring and drilling may also reduce costs. Increased bit wt gives greater rates of penetration if the mud circulation is adequate, but this is apt to lead to crooked holes. Efforts must therefore be made to stabilize the drilling string. The other possibilities are jet bits, and combined percussion and rotary action. G. D. H.

3015. Labour (productivity) standards in percussion drilling. L. Szefer. *Nafta*, 1951, **7**, 264–7.—The present state of drilling technique in Poland is below that of Roumania or U.S.S.R. Monthly rate is 120 to 150 m, and there are few signs of improvement. Time and motion study in U.S.S.R. showed the way to improvement. At present only one-sixth to one-fifth of the time is utilized on drilling operations. Cost per m tends to exceed plans. A rigid timetable will improve progress, and such timetable based on experience in similar geological formation, using similar equipment, should be presented to the crew for discussion, amended, and passed to be closely followed. M. S.

3016. Activities of drilling establishment. Anon. *Bull. Main Petrol. Inst.*, **5** (1) (Suppl. to *Nafta*, 1951, **7**), 19–20.—Brief notes on labour (productivity) standards, sliding bits, muds research, acidification of boreholes and sulphur estimation in gases by colorimetry. This method is fast and accurate to 10^{-6} g H_2S/cc of gas. M. S.

3017. What is a crooked hole? R. B. McCloy. *World Oil*, Apr. 1952, **134** (5), 156. (*API Divn of Production, Shreveport and Wichita, Mar.* 1952.)—It is generally agreed that contractural clauses, as now phrased, relating to drift in wells, do not wholly cover the requirements for control. Typical agreement clauses are quoted, with reference to State specifications. In general, it may be concluded that specification of angle of drift is not in itself sufficient, but that directional control should be included; the horizontal deviation may be appreciable. A. J. H.

3018. Gas drilling technique a success. G. M. Wilson. *World Oil*, Sept. 1952, **135** (4), 147.—The gas drilling technique has been widely applied in the San Juan Basin area because of its numerous advantages over other methods. The technique was compared to the drilling of shot holes in arid regions, and overcame the difficulties of contamination of producing zones by mud, loss of gas when drilling by cable tool, and in general, the fishing operations so often resulting from cable tool drilling. Of major importance, too, was the substantial increase in rate of penetration. Adapting rotary drilling equipment presents few problems, and specially designed diamond bits have also been successfully used. A few precautions have to be taken to suppress the fire hazard, but few cases have been attributed directly to the use of this technique. A. J. H.

3019. Open hole cement plugs. B. Barkis. *World Oil*, June 1952, **134** (7), 134.—Irrespective of the nature of the operation, whether for directional drilling or for abandonment, the importance of an efficient cementing operation cannot be overemphasized. The plug may be located in a clean hole, a hole caked with mud, or one having a mud ring, the latter resulting in an unsatisfactory type of cement plug; in general, the plug should not be opposite a shale zone, as this usually encourages the formation of a mud ring. The use of scratchers is recommended; these may be of the rotating or of the reciprocating types. References. A. J. H.

3020. Diamond coring increased in California. H. H. Stanier. *World Oil*, July 1952, **135** (1), 128; Aug. 1952, **135** (3), 167.—Although full records are not available, it is estimated that the use of diamond coring equipment in California has increased 360%. The local conditions are necessarily the final factor in the selection of type of diamond bit, but in general, these bits are economical only for hard formations. A brief introduction to coring in California and the types of bit used are given.

Apart from the size of the core-head, important features include: 1. Shape of crown; a fully rounded head has been found to give a straighter hole than the square type, but this is still largely a matter for local study. 2. Waterways; although generally most bits have between eight and twenty waterways, the number may vary in fact

between zero and thirty-two. 3. Pattern of diamond setting; most diamonds are set with two-thirds in the matrix, but the practice of setting them flush is recommended for the gauge diamonds, as these then are not damaged when running in and out of hole. The diamonds should overlap, and a higher strength is necessary at points of greater wear. Certain types of diamonds are more easily salvaged than others.

In selecting the core head it is important that the overall cost be considered. Thus a better head may permit more ft/round trip, greater speed of cutting, and a higher salvageable value; rig time must be included in the estimate. Having selected the core head, it is necessary to develop the remainder of the assembly to give at least as efficient service. Several forms of core barrel have been designed, and are available for the variety of conditions.

Comparisons of diamond coring to conventional coring are made with reference to initial capital, rig expense, and overall efficiency of operation. Six references.

A. J. H.

3021. Effect of calcium sulphate on properties of a bentonite suspension. W. E. Bergman. *World Oil*, Apr. 1952, **134** (5), 170; May 1952, **134** (6), 122.—Pt I. An extension to the work on the effect of sodium chloride on various simulated drilling fluids, which is devoted to a similar study with calcium sulphate. Materials used were bentonite, of commercial standard, quebracho, sodium hydroxide, and pure calcium sulphate. After preparation of mud samples the effect of the sulphates was determined. Further tests were made on treated muds.

The experimental procedure is described, and data are given, graphically and in tabular form. References.

Pt II. Extensive data are presented graphically, and the mechanism of the contamination of the bentonitic suspensions by the pure calcium sulphate discussed with reference to the data. References. A. J. H.

3022. New additive for control of drilling mud filtration. R. A. Salathiel. *J. Petrol. Tech.*, Mar. 1952, **4** (3), *AIMME Tech. Paper No.* 3282, 85–90.—Organic colloids, such as starch, natural gums, and altered celluloses, are used to reduce filtration rates, and this is especially true when the muds have large amounts of salt or magnesium or calcium ions causing coagulation. Starch is relatively cheap, but along with the gums is subject to fermentation, tends to increase the viscosity, and is needed in large amounts. Carboxymethylcellulose is effective, in smaller amounts, does not increase the viscosity so much as starch, but is expensive and may ferment. A water-soluble condensation product of sulphonated phenol and formaldehyde (SPIF) has proved a promising additive. The manufacturing cost is not known, but the raw materials are of moderate price. The mechanism by which the additive acts is believed to be one of plugging small openings between mineral grains in the filter cake. When the clays are coagulated the pores are large. SPIF does not appear to be absorbed on the mineral particles. In a neutral fresh-water mud it was as effective as starch in reducing filtration rate. SPIF is available as the sodium or calcium salt. It has little effect on visc or gel strength in a salt water mud, whereas starch caused increases, but was broadly similar to SPIF when used with caustic soda and pasted. The effects of SPIF on saturated salt muds were also studied, while the stability was dependent on temp, there being some increase in water loss with time.

In a field test the material was found to be easy to handle and mix, and there was no sign of deterioration of the SPIF in twenty-seven days. G. D. H.

3023. Mud control demands co-operation. A. A. Wukman. *World Oil*, July 1952, **135** (1), 121.—Since the supervisory staff cannot at all times be at the rig, it is suggested that a more extensive understanding be developed amongst the personnel. In addition to the fact that not all details are reported in an emergency, frequently the field personnel have to act on their own initiative. The complete picture may not be appreciated initially, and it is recommended that an exchange of information takes place during discussions and instructional periods. A. J. H.

3024. CMC blended for improved resistivity. W. B. Colvin. *World Oil*, June 1952, **134** (7), 140.—The difficulties encountered in the drilling of deep wells were overcome largely by the use of lime-based muds, which were not much affected by the higher temps. It has since been shown, however, that these muds tend to flatten the S.P.

(Self Potential) logs, and generally to hamper the interpretation. This was particularly undesirable in wildcat areas. The use of CMC (a sodium carboxymethylcellulose system) improved the S.P. curves, while assisting to maintain a stable mud.

Two electrologs taken in adjacent wells, one drilled with lime-based muds, the other with the CMC system, illustrate the flattening effects. A. J. H.

3025. Meter aids exact mud control. J. C. Melrose and W. B. Lilienthal. *World Oil*, July 1952, **135** (1), 136. (*AIME Petroleum Branch Fall Mtng, New Orleans.*)—The uses and applications of drilling muds are rapidly passing out of the scope of " rule of thumb " methods, and greater control is now required. The theory of plastic flow has been satisfactorily expressed in equations developed by Bingham, Buckingham, for laminar flow, and by Fanning for turbulent conditions. It has been shown that the Stormer " viscosity " is merely an apparent value defined by the Stormer rotational speed of 600 r.p.m., but such a definition overlooks the remainder of the properties, as in the equation $\mu_a = \mu_p + (B/Z)\phi$, where μ_a = apparent visc ; μ_p = plastic visc. ; B = viscometer constant ; Z = rotational speed ; ϕ = yield value. The requirement was therefore for an instrument suitable for the determination of visc at variable speeds. Such characteristics were found in the Fann V-G Meter.

The instrument was used for correlation with Stormer visc, and in the application to chemical treatment, and to pressure drop in drill pipes. Data and references.

A. J. H.

3026. Minimizing oil well cementing failures. G. B. Mangold. *World Oil*, May 1952, **134** (6), 112.—Recent investigations into the development of a suitable cement additive, have resulted in the use of a superior "pozzolan." In order adequately to cover the conditions to which the cement is subjected, a preliminary study was made of the controlling factors, under estimated deep-well conditions of up to 360° F temp and 8000 p.s.i. pressure. The compound, known as Peacoide, has recently been used in certain fields in California, and may be used as a replacement for up to 25% by wt of cement.

Factors considered include slurry wt, yield of cement, workability, bleeding, strength, behaviour under formations, setting conditions, and uniformity of set. Case histories have not yet been prepared, but initial operations appear to be highly satisfactory.

A. J. H.

3027. New process prevents cement contamination. P. Menaul. *World Oil*, Aug. 1952, **135** (3), 202.—The purpose of cementing casing is the anchoring of the string and its protection against undesirable formation fluids, such as brine, especially when under pressure. The effect of mud in the hole is to prevent the efficient bonding of cement to casing, and, owing to the contamination of the cement by mud and its subsequent increased visc, to induce channelling ; the two factors contribute to the failure of a cementing operation.

It has been shown that when a fluid is displaced by another of differing visc, the extent of mixing is dependent on the ratio of viscosities, there being no chemical interaction ; when the driving fluid is the more viscous, little mixing occurs, and vice versa. The new process consists therefore in pumping ahead of the cement, a fluid plug of non-reactive substance of visc greater than mud, and less than cement. The function of the plug is to displace mud from the annular area, without itself gelling up, as with cement-cut mud, and to be displaced similarly by the cement. When this hardens, it is then more uniform. References. A. J. H.

3028. Lost circulation corrective : time-setting clay cement. J. U. Messenger and J. S. McNeil. *J. Petrol. Tech.*, Mar. 1952, **4** (3), *AIMME Tech. Paper No.* 3283, 59–64.—The control and prevention of lost circulation in drilling oil wells is a frequent problem. In the past this has been tackled by adjusting the mud density, by adding fibrous or flaky material to cause bridging, or by cementing. The new technique employs an aqueous slurry of specially selected clay, a smaller amount of aluminium silicate to increase the strength of the set clay cement, and a retarder to increase the pumping time of the slurry. It is pumped down the pipe and spotted against the loss zone and then squeezed that into zone. After a relatively short period the gel strength of the slurry increases greatly forming a plastic material with

sufficient strength to seal the zone against mud losses during further operations. It is possible to treat longer zones in one operation than can be done with cement. The plug can be drilled more rapidly than in the case of cement, and there is a shorter waiting period. There is no appreciable impairment of the mud, and the low water-loss of the clay cement means that there is little damage to the formation by filtrate.

Large volumes may be needed to seal zones of serious loss, especially where temp are low, and the strength is less than for cement.

Among twelve cases of the use of time-setting clay cement ten were successful. The amounts placed ranged 14 to 522 brl, with slurry densities 10·8 to 11·4 lb/gal.

G. D. H.

3029. Common fallacies in electric log interpretation. R. G. Hamilton. *World Oil*, Apr. 1952, **134** (5), 141.—If carefully analysed, electric logs provide reliable data on formation characteristics. Common fallacies and step-by-step procedure are given to assist the non-specialist to an understanding of the log interpretation. A. J. H.

3030. Quantitative aspects of electric log interpretation. J. E. Walstrom. *J. Petrol. Tech.*, Feb. 1952, **4** (2), *AIMME Tech. Paper No.* 3280, 47–58.—The electric logs can be used for quantitative determinations of water saturation of sands or sandstones in development wells if the formation factor and water resistivity are known, and provided that the clay content of the formation and its effect on the electrical behaviour are also known. The determinations are unreliable in thinly bedded sand–shale sections. Cores of the producing sand will give information on the formation factor and clay content.

In exploratory wells the electric log aids in selecting those formations with evidence of containing oil or gas, and then the formations can be tested. Care must be taken to avoid missing possible production from fractured formations. The log interpretation is then qualitative, but of considerable value.

Research and development are proceeding, and it is possible that improved methods will make it possible to obtain a more accurate measurement of the formation factor from invaded zone measurements, while better determinations of interstitial water resistivity may be practicable from the potential curve. G. D. H.

3031. Scintillometer used in radiation logging. L. W. Toelke. *World Oil*, Aug. 1952, **135** (3), 143.—The advantages of this instrument, which has already received much attention in nuclear physics, include high efficiency; no instrument drift; sensitive recording of thin beds; records made on original sheet and can be examined during the test; and easy interpretation. The radiation log is a measure of the natural radioactivity of the earth; shale beds have high radioactivity, and sands, limestones, dolomites, and salts have low radioactivity. Depth control and speed of running are good. Research continues for the further development of the method. A. J. H.

3032. Guarded electrode system improves interpretation of Lower Wilcox. R. C. Howard and R. H. Winn. *World Oil*, July 1952, **135** (1), 170.—The interpretation of conventional logs taken in the Lower Wilcox sand had been much complicated by the erratic lithology, and necessitated a large number of formation tests to select suitable producing zones; lignitic streaks are interbedded in the sand. The disadvantages of the conventional log are due to these streaks, and to the invasion of the mud filtrate. A study of the conditions revealed that the use of a guarded electrode run with low resistivity and low water loss muds would greatly improve the definition of the log. This is substantiated by logs reproduced, showing the comparison. It was recommended that normal drilling procedure be carried out to just above the producing zones, when a change-over to a starch-organic mud be made, and after drilling, the zones surveyed by the above method. This has resulted in a 16% saving on general well costs. References. A. J. H.

3033. Science and technique in Soviet Union. Small diameter drilling equipment with hydraulic string feed. R. Piatkowicz. *Nafta*, 1951, **7**, 281–6, 315–17 (" *Boorovoye Dyelo*," *by Vozdvizhenski and Volkov*).—Describes in outline the transportable base on which the motor, the hydraulic feed, and gearbox are fitted. Detailed description of

rotary table and hydraulic feed follows with many diagrams. Planetary and differential drawworks and gearbox are also described and formulæ given. Drilling equipment incorporating all above features and suitable for depth of 1200 m is produced under code name ZIF 1200. This is also described.

A short anonymous note on p. 287 describes Roumanian tool for removing cement from inside of pipes after formation cementing. M. S.

3034. Mill rejects pass tests. H. G. Texter. *World Oil*, May 1952, **134** (6), 99; June 1952, **134** (7), 144.—Pt 1. A report on field tests using pipe normally rejected by mills because of manufacturing defects; some of the wells drilled with such pipe have been taken through salt formations using underweight on drill collars to increase the bending stresses in the pipe, and also using the recommended number of drill collars. In general, it may be said that a greater proportion of " mill failures " than was formerly thought advisable could be utilized in the field. Some photographic examples are given. Pt 2. In order to separate the effects of corrosion from those failures due to mechanical weakness in the pipe, steps were taken to minimize corrosion. The results are summarized, and in conclusion the limit to which inside defects may be permitted has been established, but the external effects have not been fully tested. In general, fishing operations were not the cause of failures. A. J. H.

3035. Shot peening cuts drill pipe failures. R. Irving. *World Oil*, July 1952, **135** (1), 109—Increasing the life of drill pipe is particularly important at a time when materials are short, and also for the drilling of deep wells. Two preventive measures against failure are sand-blasting and shot peening. These may be applied separately or as a dual process, but the recommended practice is to clean the pipe by sand blasting and then to shot-peen the pipe for the relief of tensional stresses within the material. Sand blasting is useful for the prevention of localized corrosion, and subsequent to shot peening the surface becomes more resistant to general corrosion. It is claimed that the combined process enables the material to withstand up to four times the normal number of stress cycles. A. J. H.

3036. Savings and improvements in the management of drilling pipes in the (Polish) 6-year plan. J. Pawlowski. *Nafta*, 1951, **7**, 246–9.—In Pt 1 author describes procedure to prolong life of pipes and in Pt 2 points to the need for pipe-budgeting and advanced planning to avoid bottlenecks. M. S.

3037. Fibercast plastic pipe finds use as oil well tubing. T. D. Gardner. *World Petrol.*, Aug. 1952, **23** (9) 42–4.—Fibercast is a thermosetting resin with good structural strength and heat stability between 65° and 300° F. Installation and use in a salt water disposal well of 3½-inch O.D. tube is described. Laboratory tests show tensile strength of assembly, including threads, couplings, and glue lines, to be 7000 lb, whilst tubing itself had strength of 10,000 lb. String weighing 7000 lb was lowered into well giving test of full capacity. Tubing is available in 300 p.s.i. internal and 500 p.s.i. external pressures. Weights vary from 17 lb/20 ft of 3-inch line to 50 lb for 4½ inches. Prices higher than for steel pipe of comparable size, but installed prices usually lower. Advantages include chemical inertness to most common corrosive materials and lightness (one-fifth of wt of steel). E. B.

3038. Diesel-electric drilling barge " G.P.2." Anon. *Engineer*, 1952, **194**, 425–6. —To enable drilling operations to be carried out in the deeper waters of Lake Maracaibo, Shell Petroleum Co. is building a self-contained diesel-electric power barge of 2545 tons displacement. The barge is non-propelled, 156 ft long, 80 ft broad, and has a draught of 7 ft 6 in. It is equipped with four main 450-kW D.C. generators and two auxiliary generators, all diesel engine driven. There are two main mud pumps suitable for 3500 p.s.i. pressure driven by 850-h.p. motors and a multi-cyl 300-h.p. pump for cementing operations suitable for 10,000 p.s.i. pressure. Motors are provided for all drilling operations, and in conjunction with a permanent piled steel structure at the site of each well, the barge is capable of drilling to 15,000 ft at points 15 miles or more from the shore. Sufficient stores, etc., are carried for an endurance of thirty days. A. C.

3039. Patents. U.S.P. 2,590,880 (30.12.49; 1.4.52). L. C. Miller. A device for determining the orientation of a tool supported by a pipe extending into a well bore.

U.S.P. 2,590,892 (20.9.46; 1.4.52). A. B. Long, assr to Sun Oil Co. Apparatus for concurrently measuring and recording temp, pressure, and visc of well borehole liquids.

U.S.P. 2,590,931 (11.2.49; 1.4.52). B. A. Cabaniss, assr to Sperry-Sun Well Surveying Co. Chemically heated apparatus, including radially extending knives in staggered arrangement for heating and removing a paraffin deposit from the inside wall of a pipeline.

U.S.P. 2,591,087 (10.3.47; 1.4.52). R. E. Millican. A bottom hole hydro-pneumatic graduator for oil wells.

U.S.P. 2,591,488 (8.11.46; 1.4.52). L. Yost, assr to A. O. Smith Corpn and California Research Corpn. Balanced turbodrill for well drilling.

U.S.P. 2,591, 599 (25.8.49; 1.4.52). M. H. Parks, assr to Standard Oil Development Co. Structure for drilling wells in deep water.

U.S.P. 2,591,603 (25.7.46; 1.4.52). T. M. Ragan, assr to Baker Oil Tools Inc. Plug catcher and indicator.

U.S.P. 2,591,737 (28.11.50; 8.4.52). R. E. Souther, assr to National Lead Co. Detecting oil in mud-laden well drilling fluids by steam distillation of the fluid.

U.S.P. 2,591,807 (23.8.47; 8.4.52). H. M. Greene. Oil well cementing.

U.S.P. 2,592,378 (30.10.48; 8.4.52). P. E. Chaney and W. E. Barnes, assrs to Sun Oil Co. Apparatus for the sonic determination of zones of lost circulation of drilling mud in well boreholes. R. C. R.

Production

3040. Engineering study of the Cook Ranch field, Shackelford County, Texas. W. W. Wilson. *J. Petrol. Tech.*, Mar. 1952, **4** (3), *AIMME Tech. Paper No.* 3285, 77–84.—The Cook Ranch field was discovered in 1926, and produces from a lens in the Cook sand of Lower Permian or Upper Pennsylvanian age at depths of about 1300 ft. The average dip is 50 ft/mile to the northwest. The oil zone occupied 14,512 acre-ft, and the gas cap 1000 acre-ft originally. Air permeabilities range 50 to 2300 mD, and porosities 12·5 to 28·2%; interstitial water was about 10%. The solution G.O.R. may have been 400 cu. ft/brl, and the original formation volume factor 1·2. The original stock-tank oil content of the reservoir is estimated as 20,273,264 brl. Gas injection began in July 1927. Very full records have been kept. Fifty-six wells have been used for injection, not more than twenty-nine at once. Cumulative injection volumes for single wells range 1000 M.c.f. to 1,400,000 M.c.f. To the end of 1950 the cumulative crude recovery was 14,701,131 brl; in addition, 46,558,446 gal of natural gasoline has been recovered from the produced gas. Cumulative gas injection was 27,481,295 M.c.f.

Details of the performance of various areas and of the whole field are given. A recently drilled well had 18% of oil in the upper 27 ft and 40% in the bottom 5 ft of the sand, and the well when last reported was giving 20 b.d. with a G.O.R. of 5300 cu. ft/brl. Gravity drainage appears to have contributed extensively to the oil recovery. Initially the gas may have displaced the oil mainly horizontally, giving a low G.O.R., but after break-through vertical movement became prominent. G. D. H.

3041. A discussion of petroleum engineering problems in the Burgan field, Kuwait. L. G. May. *Proc. Third World Petrol. Congr.*, 1951, **2**, 697–700.—The early history, geology, and development of the Burgan field in Kuwait are outlined, and current exploitation methods are briefly described.

Much research has been carried out on the taking of bottom hole crude samples and their analysis in P.V.T. equipment. The crude in the main pay zones is generally undersaturated, and the average producing G.O.R. is approx 500 cu. ft/brl.

Over 250 million brl have been produced from Burgan since June 1946, when the first tanker was loaded. Ninety-eight flowers had been completed to Oct. 1950, with a field potential of approx 500,000 b.d. C. A. F.

3042. Techniques in the Kirkuk oilfield. H. A. Freeman. *Proc. Third World Petrol. Congr.*, 1951, **2**, 683–95.—The nature and methods of production of the Kirkuk limestone reservoir in Iraq are described, together with the methods developed to minimize pressure losses in long flow lines.

Accurate measurements of oil–water contact in observation wells are considered important, but are often masked by drilling water lost to the formation. This difficulty may be overcome by drilling with oil in one of several ways; normal circulation of degassed crude, by the "balanced column" method in which the drilling fluid is diluted with petrol so as to give a mixture of the same density as the reservoir fluid, and by the method of "reversed flow." The method of measuring the depth of oil–water content in observation wells is described. C. A. F.

3043. The Cretaceous limestone producing areas of the Mara and Maracaibo districts, Venezuela. M. Stephenson. *Proc. Third World Petrol. Congr.*, 1951, **2**, 665–81.—The reservoir characteristics and production engineering aspects of the Cretaceous limestone fields in the Mara and Maracaibo areas of Venezuela are described. These fields have large volumes, thick oil columns, but low intergranular porosity. Productivity is controlled by widely distributed fissure systems.

There has been rapid development of the fields, and care has been taken to conserve their natural energy. C. A. F.

3044. The Poza Rica field. J. Colomo and A. Barnetche. *Proc. Third World Petrol. Congr.*, 1951, **2**, 647–63.—The Poza Rica field, approx 110 miles northeast of Mexico City, was discovered in 1930, following surface and geophysical mapping. The structure is 11 miles by 5 miles wide, with a steep northeast flank and an almost flat southwest flank; pay is Cretaceous Tamabra limestone at approx 2000 m subsea, and the productive area is limited to the northeast, northwest, and southeast by brine, and to the southwest by lack of porosity. (Porosity appears to be directly related to dolomitization.)

The productive interval is divided into three zones, the upper and lower being fairly consistent throughout the field, the middle being erratic.

Porosity and permeability of cores from parts of the field have shown the porosity to be mainly intergranular and the permeability to vary widely, laterally, and vertically.

Probable virgin bottom hole pressure was 3390 p.s.i.g., and average reservoir temp is 204° F. An interstitial water content of 13·5% is used in calc reserves. The original gas/oil level was at 2030 m subsea, and probable oil/water level 2270 m subsea. It is suggested that the oil/water level was not horizontal when the field was discovered, but that it was still undergoing gravitational segregation.

The original oil in place has been computed as 4·78 billion brl, and a method has been developed to apply the material balance equation to the field; this is described in detail.

The reservoir performance is predicted for the near future under various gas and/or water injection projects.

The handling of the oil and gas in the field is briefly outlined.

Five references. C. A. F.

3045. Good data necessary for good reservoir engineering. J. A. Murphy. *World Oil*, July 1952, **135** (1), 194. (*Spring Mtng, Pacific Coast District, API Divn of Production, 15 May, Los Angeles.*)—Research into improved methods of production and control is wasted unless followed by the accurate recording of production and reservoir data. The "material balance" equation, for example, depends on numerous variables, of which certain are field values. The basic factors and the producing mechanisms are discussed. A. J. H.

3046. Improved multiphase flow studies employing radioactive tracers. V. A. Josendale, B. B. Sandiford, and J. W. Wilson. *J. Petrol. Tech.*, Mar. 1952, **4** (3), *AIMME Tech. Paper No.* 3284, 65–76.—Two radioactive tracers have been tested as a means of determining core saturation in multiphase flow studies. Cæsium chloride, a water-soluble tracer, caused difficulties in low permeability cores because cæsium was sorbed by the core or water-wet pads. Iodobenzene was satisfactory as an oil-phase tracer. It was insoluble in water and not sorbed by the core materials used.

The tracers were used in experiments on the saturation profiles during capillary and dynamic de-saturations and relative permeability measurements in oil–water and gas–oil systems. The results of these experiments are presented. The mobility of brine and of the oil phase was studied. In the former case it was shown that diffusion played no significant part. For oil the degree of mobility is markedly affected by the pore size distribution. G. D. H.

3047. Depletion recovery calculations simplified. R. W. Hillyer. *World Oil*, Sept. 1952, **135** (4), 226.—Estimation of recovery of gas and condensate may be made by physical tests of the reservoir fluids, by mathematical calculation, or by comparison with similar reservoirs. This method consists in determining the hydrocarbons contained within a unit volume before and after depletion, and thus estimating the overall recovery. The method is described step by step. The method assumes no fluid invasion. A. J. H.

3048. Gas injection in the Anton-Irish field of west Texas. Anon. *World Oil*, Apr. 1952, **134** (5), 220.—The Anton-Irish field was discovered in 1944, and produces oil from the Clearfork Limestone at an approx depth of 5900 ft with a well spacing of 40 acres/well. It was shown in mid-1948 that the oil had a low gas content of 148 cu. ft/brl, and as the principal sources of energy were fluid expansion and dissolved gas, it was decided to commence a gas injection project. Gas was piped from the El Paso Natural Gas Co. and compressed from approx 700 to 1500 p.s.i. for injection into, initially, twelve input wells. The allowable production rate per well has been increased from 42 to 100 b.d. A. J. H.

3049. High pressure gas injected in Block 31. B. L. Griffith and W. M. Hollrah. *World Oil*, July 1952, **135** (1), 163.—A more detailed study of the gas injection project of the field in Crane County, Texas, in which several operators have combined to ensure a more efficient system. The study has been augmented by lab details; an estimate of 77% sweep is given following potentiometric model tests (*cf.* Abs. 1325). A. J. H.

3050. Application of gas lift to everyday production problems. E. D. McMurry. *World Oil*, May 1952, **134** (6), 151; June 1952, **134** (7), 206.—Pt I. Gas lift is finding wide application because of its economic method of producing oil, and because of its general flexibility; the range of producing rates has been stated to be between 3 and 20,000 b.d. The preliminary investigation should include a survey of the completion methods of the well and their suitability to such a producing method, and an estimation of the cost of the gas, and its general processing, and also the practicability of installing equipment which must necessarily involve an appreciable capital outlay. In general, the characteristics of the oil do not detract from the advantages of the method.

As the most common type of valve in use is the pressure-charged valve, reference is made to four such valves only; they function as back pressure regulators, and are generally installed on mandrels. Five groups of wells are selected for analysis, of which two are considered in the first part. These wells may be divided into four groups covering the range of high bottom hole pressure and high P.I. (productivity index), to low bottom hole pressure and low P.I., and in the fifth case, dually completed wells. These groups account for about 99% of the total gas lift wells.

After analysis of the conditions normally prevalent in the group, a typical example is taken, and the spacing of valves estimated from the formula first proposed by the author in 1946.

Pt II. The two groups covering low bottom hole pressure, and high and low P.I. are described. Gas lift in dual completions is also satisfactory. Installations vary, but, in general, the gas may be introduced in the annular area between a macaroni string and tubing, disadvantages of which are low max gas throughput and limited operating depth of about 3500 to 4000 ft; or a tandem assembly, in which large vol rates are possible, may be used. A. J. H.

3051. Getting most from rotative gas lift. B. Moore. *World Oil*, July 1952, **135** (1), 181.—Substantial savings may be effected by considering the surface compressors and ensuring that suction and discharge pressures and throughput vol rates of gas have been

selected for optimum operation. An eight-well gas lift lease is chosen as an example of a compact unit. Hydrates present some problems in certain fields. A. J. H.

3052. Information on Hydrafrac process. J. C. Padgett. *Proc. Third World Petrol. Congr.*, 1951, **2**, 618–19.—Hydrafrac is a process for fracturing a pay zone by means of hydraulic pressure in order to obtain a greater effective permeability around the well bore. The fracturing is accomplished by pumping into the zone gels which produce a frictional resistance and allow hydraulic pressure higher than the natural shear strength of the formation to be applied.

The factors to be considered in applying the Hydrafrac process are outlined; these are: the state of depletion of the well; formation permeability; formation thickness; prior workovers; and isolation of formation.

Tests on 316 wells treated showed 72·5% success with an average production increase of 45·8 b.d. C. A. F.

3053. A study of the permanence of production increases due to hydraulic fracture treatments. C. R. Fast. *J. Petrol. Tech.*, Feb. 1952, **4** (2), 10.—Data from sixty-five Hydrafrac-treated wells in twenty-six fields have been analysed. At the end of six months forty-five out of sixty showed sustained production increase. At the end of two years fifteen out of twenty-five showed sustained production increase; at the end of three years ten out of eighteen had a greater production rate than before Hydrafrac treatment. For those with sustained increase, after two years the effective average rate was 100% above the pretreatment level; after three years the rate was 34% above the pretreatment level. G. D. H.

3054. Completing wells in the Gulf Coast Sands. J. E. Kastrop. *World Oil*, June 1952, **134** (7), 194.—The former attitude of emphasizing more the drilling of the well, than the other features, has now been superseded by having more regard for the conservation of energy within the reservoir. Consequently, completion practices are of major importance. In this area many of the producing formations are made up of sand bodies interbedded with shale; depending of the relative position within the reservoir, these have a tendency for "gas fingering" and "water coning." These difficulties have been overcome by a re-arrangement of the perforating programme. Gravel packing is used for the exclusion of sand particles, while the consolidation by plastics is also practised. A. J. H.

3055. Core analyses aid Wilcox completions. B. A. Elmdahl. *World Oil*, June 1952, **134** (7), 181.—Core analyses were made on the Wilcox formation in the early stages of the development, and since the sand exhibits characteristics which are of particular importance to such analyses, a general discussion on technique is given. Samples were taken from the interior portion of the diamond core in order not to test the flushed vol; the extent of flushing action is discussed. Basically methods are similar to other fields. Much depends on the depth from which the core was taken and the characteristics of the reservoir fluids.

The discussion is continued in respect to particular wells in the Wilcox area. Data are given. A. J. H.

3056. Note on the X-ray absorption method of determining fluid saturation in cores. T. M. Geffen and R. E. Gladfelter. *J. Petrol. Tech.*, Mar. 1952, **4** (3), 15.—In the experiments the absorbing liquid was a solution of about 5% by vol of iodobenzene in a close-cut hydrocarbon fraction, or a solution of 50,000 p.p.m. of sodium iodide in water. In one form of experiment the liquid was inside the core, in the other it was outside. In the former case the saturation range covered that encountered in drainage and inbibition. In the latter case the core contained only gas but various thicknesses of oil were placed in front of it in plastic cells. For drainage, inbibition, and the case of liquid outside the core, a linear relationship was found for liquid saturation or thickness and the logarithm of the relative X-ray transmission. There was no evidence of the non-unique relationship suggested by Lipson, although for flow there was hysteresis. The explanation may be that Lipson considered conditions in very small groups of pores, whereas in practice the X-ray beam is affected by very large numbers of pores. G. D. H.

3057. Laboratory experiments on the displacement of oil by water from packs of granular material. W. F. Engleberts and L. J. Klinkenberg. *Proc. Third World Petrol. Congr.*, 1951, **2**, 544–54.—The displacement of oil by water is described for model experiments using packs of unconsolidated and approx uniform grains, in which the experimental conditions attempt to simulate water drive in natural reservoirs.

It is concluded from these experiments that it is not to be expected that changes in capillary forces will affect appreciably natural water drives and that the higher the visc of the oil the lower is the rate of displacement which is effective in reducing the amount of water produced with the oil. Flood pot tests can be useful in providing information on the residual oil content in the flooded areas if the sand contains connate water.

Four references C. A. F.

3058. Apparent and actual minimum water content of oil reservoir rocks. W. Ruehl. *Proc. Third World Petrol. Congr.*, 1951, **2**, 522–6.—It has been found that the water content of sands taken from the Wietze field in Germany is two to three times less than the content found by capillary pressure tests of the irreducible interstitial water content; it is concluded that the capillary pressure methods which are widely used possibly cannot reproduce the reduction in the water content to the extent which might obtain in Nature over geological time. C. A. F.

3059. The exploitation of oilfields of extremely high oil-viscosity by wells, under the application of thermal energy. A. K. Winkler. *Proc. Third World Petrol. Congr.*, 1951, **2**, 528–42.—A technique has been developed which enables crude oils of very high visc, $\eta > 70{,}000$ cp at 20° C, to be produced economically; such oils cannot be produced by conventional methods.

The technique has been introduced in the Leoprechtung field in Austria, and consists of heating the formation by the addition of small quantities of steam in conjunction with an air lift, or by electric heating devices. The temp increase in the formation results in an increase in production rate, a higher total well production, and a better ultimate recovery for the field. Drilling costs are also reduced since fewer wells are required.

Production curves are included. C. A. F.

3060. Efficiency of gas displacement from porous media by liquid flooding. T. M. Geffen, D. R. Parrish, G. W. Haynes, and R. A. Morse. *J. Petrol. Tech.*, Feb. 1952, **4** (2), *AIMME Tech. Paper No.* 3279, 29–38.—Gas recoveries of 80 to 95% have commonly been assumed for water-drive fields, because of the low visc of gas, and the erroneous assumption that, since gas as a displacing agent can flow at gas saturations of 1 to 15%, the flow relationship will be the same when gas is being displaced. Water flooding an oil reservoir normally leaves 15 to 50% of oil.

In investigating the efficiency of gas displacement by water the laboratory work included relative permeability tests, gas displacement by water on 1-ft cores cut parallel to the bedding, water-flooding a 16-ft sandstone core in which the initial water distribution was attained by gravity drainage, and gas displacement from dry core plugs by water inbibition. In the field the gas saturation of a watered-out gas sand was obtained by using a pressure core barrel to take a section of the 4500-ft gas sand of the West Beaumont field, Texas.

In the 1-ft cores the residual gas saturation was 28·7 to 36·5% when the pressure, temp, and rate of water advance were varied.

Flooding of the 16-ft sandstone column, which simulated a gas reservoir, gave a residual gas saturation of about 34%. The inbibition tests gave saturations of 31·8 to 35·5%. The cores from the pressure core barrel gave gas saturations of 16·7 and 18·5%.

The results as a whole indicate that the residual gas saturation after water flooding may be 15 to 50%, and hence similar to the residual oil saturation left in water-flooded oil sands. It was also concluded that small core plugs are suitable for laboratory measurements of residual gas saturations. G. D. H.

3061. Displacement mechanism in multi-well systems. L. R. Kern. *J. Petrol. Tech.*, Feb. 1952, **4** (2), *AIMME Tech. Paper No.* 3220, 39–46.—A method has previously been described for calculating the saturation distribution behind the interface between

the displaced and displacing phases in a gas-injection or water-flooding operation. This involved graphical integration. The method has now been extended to a more general situation, with analytical integration. The reservoir may initially contain flowing quantities of the displacing fluid, or it may be above saturation pressure, so that solution effects are present. There may be production behind the interface, as when a well is produced after break-through of the displacing fluid. In the method the displacing phase fraction is represented by an empirical equation, resulting in simple expressions for the producing gas/or water/oil ratio and cumulative production.

A numerical example illustrating the new method is given with detailed working.

G. D. H.

3062. Secondary recovery of oil in California. A. G. Loomis and G. B. Shea. *Proc. Third World Petrol. Congr.*, 1951, **2**, 608–16.—The broad aspects of secondary recovery in California are discussed. Most of the current operations are of the pressure maintenance type, in which the reservoir pressure is maintained by injecting natural gas, but the possibilities of recovery from stripper fields is also being investigated by gas drive and water flooding.

The economic value of gas injection has been proved by field operations, but the few water flood projects have not yet been proved to be economical; more field data would be valuable in this respect.

Problems of gas injection and water flooding in California are described and projects are listed.

Twenty-three references. C. A. F.

3063. Flooding ups limestone production. M. H. Durbrow, H. L. Temple, and W. E. Landrum. *World Oil*, May 1952, **134** (6), 166.—Little has been published on the secondary recovery of oil from near-depleted limestone or dolomite reservoirs by water flooding. A pilot project was first tried and proved satisfactory, and subsequently, the project was expanded to include the entire pool.

The general history of the pool and its development are given. It is concluded that such a method may be used only where the zones around the injection well are in permeable connexion with those around the producing wells; in the case of fractured limestone reservoirs, it is unlikely that the invasion would be complete.

References. A. J. H.

3064. Oil recovery by fluid injection. P. D. Torrey. *Proc. Third World Petrol. Congr.*, 1951, **2**, 565–603.—Oil recovery by fluid injection embraces secondary recovery or pressure maintenance utilizing gas or water injection; controlling factors in recovery are the physical characteristics of the reservoirs.

The effects of geological structure and porosity and permeability, and the mechanism of fluid injection are described in detail, and the manner in which the contents of the reservoir and the characters of the reservoirs themselves control the injection of fluids and the production of fluids is emphasized. Methods of evaluating the efficacy of injection operations are also proposed.

Important examples of the results obtained by injection are described, and new techniques for increasing recovery efficiency are outlined; these include the injection of inert gases, the Squires process of applying heat to the reservoir rocks, and the releasing of oil by bacteria.

Eighty-five references. C. A. F.

3065. Flowmeter for water injectivity profiling. R. G. Piety and B. F. Wiley. *World Oil*, May 1952, **134** (6), 176.—The principle of operation consists in controlling the rate of flow through a calibrated pump, and in measuring any overall change in flow rate with the aid of an anemometer set in a by-pass. Under equilibrium conditions the flow in the casing is equivalent to that in the pump, and no flow would be recorded by the anemometer. The instrument may be used for the determination of water levels, the location of casing leaks, and the logging of the well injectivity profile. A. J. H.

3066. Increase oil recovery and cut storage costs. H. T. Kennedy. *World Oil*, Aug. 1952, **135** (3), 197.—Seasonal demand for LPG introduces problems of storage. Having regard to the substantial volume of even small reservoirs, and also to the

significant effect obtained by injecting LPG into partially depleted reservoirs, the combined effects were studied. In solution-gas-drive reservoirs, a vol of 1000 to 2000 brl pore space per acre ft may be available. The effect of injecting LPG is primarily to cause an increase in oil volume, reducing its visc and increasing the relative permeability of the sand to oil. Furthermore, lab experiments indicate a remarkably high oil recovery.

An ideal reservoir is selected and reference made to the relevant equation for flow in such a reservoir, and to data of representative mixtures. The conclusion is that the method of storing LPG in these reservoirs helps to solve the dual-problem of storage and secondary recovery.

References. A. J. H.

3067. Pumping deep wells with sucker rods. K. N. Mills. *World Oil*, Aug. 1952, **135** (3), 222.—A well-balanced pumping system is essential to an efficient programme, and may be developed only after consideration of all the factors. Where possible these are inter-related by formulæ. Data required for such designs are given graphically. Particular attention is paid to the practical side. A. J. H.

3068. Crude oil dehydration : a universal size frequency distribution law for emulsion particles. N. Schwarz. *Proc. Third World Petrol. Congr.*, 1951, **2**, 628–46.—The kinetics of crude oil emulsion are briefly discussed, and a universal distribution law, by which the particle size distribution of all stable emulsions can be represented as one single function containing only one parameter, is proposed.

In this law, which is derived statistically, the parameter is six times the reciprocal surface area, and the use of the single function enables the stability of emulsions to be defined.

The law was tested on crude oil emulsions.

Thirty-four references. C. A. F.

3069. Drill-stem testing methods move ahead. J. E. Smith. *World Oil*, May 1952, **134** (6), 182.—The method of taking a sub-surface sample of oil-gas, or formation water, at the time of the drill-stem test provides for an early and accurate analysis of the sub-surface fluids.

The routines for saturated and under-saturated reservoir fluids are similar, except that with the former a period of well shut-in is recommended to permit the gas/oil interface to rise above the unit. A sample of approx 1300 cc may be taken, and is then available directly for p-v-t or other analysis. A. J. H.

3070. Solvent washing of wells in the Digboi field, Assam. H. B. Kale *et al.* *Proc. Third World Petrol. Congr.*, 1951, **2**, 621–7.—Production problems due to the high paraffin wax content of the Digboi field in Assam have been eased by injecting kerosene into low pressure wells; it is suspected that as the reservoir pressure falls wax is deposited behind the screen pipe and in the pores of the sand.

Approx 250,000 gal solvent per month are being injected, effecting an estimated increase of over 250 b.d. (in 1949). C. A. F.

3071. The application of numerical methods to cycling and flooding problems. C. H. Fay and M. Prats. *Proc. Third World Petrol. Congr.*, 1951, **2**, 555–63.—The mathematical computation of two-dimensional flow fields, used in order to determine invasion patterns in cycling and flooding problems, is discussed for single fluids and for two fluids. The potentiometric method provides an analogy for simple fluid invasion, but no analogous method has been devised for two-fluid invasion.

In the case of two-fluid invasion, assuming that the effective fluid conductivity for the displacing phase is four times that of the displaced phase, and assuming a conventional five-spot pattern, the break through recovery was calculated to be 45%, compared with 72% for single fluid invasion.

Twelve references. C. A. F.

3072. A method for predicting the tendency of oilfield waters to deposit calcium sulphate. H. A. Stiff and L. E. Davis. *J. Petrol. Tech.*, Feb. 1952, **4** (2), *AIMME Tech. Paper No.* 3278, 25–8.—A graphical method is described whereby the tendency of oilfield waters to precipitate calcium sulphate under a variety of conditions can be predicted.

The effects of temp, the presence of common ions, the sodium ion, and the magnesium ion are plotted. Each of these factors is incorporated in an equation.

The technique has been applied to the formation of scale deposits in heat treaters, boilers, etc., due to mingling of waters via casing leaks, in cooling water systems, and as a result of water injection. G. D. H.

3073. Sludge effects on oil behaviour. F. R. Cozzens. *World Oil*, Apr. 1952, **134** (5), 226.—The restriction on oil movement within a reservoir may be attributed in part to the effect of sludge; this effect is variable, and in view of the uncertainty has frequently been overlooked. The two main types are the formation sludges and the bore sludges. The well bore normally acts as a trap for sludge, and should be examined for accumulation when this is suspected. Analysis of the sludge facilitates the selection of the chemical for breaking down the emulsion of the sludge, and such treatment should be directed at the well bore region.

The methods of treating and cleaning, and the principles of prevention of sludge formation are outlined. A. J. H.

3074. Eolian sand control. R. C. Kerr and J. O. Nigra. *Bull. Amer. Ass. Petrol. Geol.*, 1952, **36**, 1541–73.—In sandy areas oilfield operations are faced with the problem of sand encroachment and accumulation, a serious hazard to equipment and a constant drain on manpower and transportation resources as well as a frequently recurring charge against operations. In recent years sand-control studies have proved that drifting sand can be effectively trapped and retained with four objectives: the destruction or stabilization of such sand accumulations as dunes, to prevent their migration to operational sites; the diversion of wind-blown sand round buildings and areas; the direct, permanent stoppage or impounding of sand before the location to be protected; and the rendering of deliberate aid to sand movement so as to avoid deposition over a given area. To achieve these objectives, transposing, planting, paving, panelling, fencing, and oiling can be employed, singly or in combination. E. N. T.

3075. Stabilizing condensate at Tigre Lagoon. E. E. Sands and A. Dawson. *World Oil*, June 1952, **134** (7), 222.—With the possibility of high transport losses through evaporation, and with a local market for the gas, it was decided to instal a stabilization plant for the condensate production. The condensate contained insufficient propane and butane for economical gasoline separation, and a low-temp separation unit was therefore installed. Initial estimates indicate a 10% increase in recovery. Flow diagram. A. J. H.

3076. Submersible barge services marshland wells in Louisiana. T. O. Mallien. *World Oil*, June 1952, **134** (7), 212.—Substantial reduction in servicing costs is effected by the use of the barge, which may be navigated in water less than 2 ft deep, and may be submerged in up to 8 ft of water. The power for manœuvring is external, but otherwise the barge is self-contained. Its scope ranges from general swabbing to the permanent abandonment of wells of up to 10,000 ft deep. The approx dimensions are 88 by 30 by 10 ft, and the barge carries a two-leg telescoping mast; working height below the crown block is 65 ft. The general layout is described, with photographs and a barge plan. A. J. H.

3077. Patents. U.S.P. 2,591,101 (29.5.46: 1.4.52). E. B. Smith and L. D. Mowrey. A replaceable pumping unit for insertion in a deep well casing.

U.S.P. 2,951,142 (16.4.48; 1.4.52). J. E. Eckel, assr to Oil Center Tool Company. Wellhead.

U.S.P. 2,591,174 (12.2.46; 1.4.52). J. N. Martin. Well pump valve for deep wells.

U.S.P. 2,592,325 (28.1.43; 8.4.52). H. C. Otis and J. C. Luccous. Well flow regulating device.

U.S.P. 2,592,511 (7.1.50; 8.4.52). J. F. Chittum, assr to California Research Corpn. Preventing deposition of impingement carbonate scale on metal parts in a well producing oil or gas and water. R. C. R.

Oilfield Development

3078. The exploitation of natural gas at Saint-Marcet. F. de Boulard. *Proc. Third World Petrol. Congr.*, 1951, **2**, 701–20 (in French).—Wet gas was discovered at St-Marcet in southwestern France in 1939, and the field has been exploited since 1942; cumulative production now exceeds 1·1 billion cu. m., or approx one-fifth of the original estimated reserves.

Methods of production and equipment are described and reservoir data presented. Installations for treating and distributing the produced gas are also described; these include the gasoline plant at Boussens and a pipeline network.

The gas is consumed by industry (½), domestic users (¼), and is also distributed as compressed fuel gas (¼). C. A. F.

3079. Petroleum in the Pyrenees. A. Coulty. *Bull. Ass. franç. Tech. Pétrole*, 31.1.52 (91), 3–22.—The R.A.P. holds concessions in the Petites Pyrenees. Study of surface structures and geophysical work led to the discovery of Saint-Marcet and Proupiary. 160,000 m has been drilled, 55,000 m being on these two structures. Fifteen out of twenty wells are productive at Saint-Marcet. One well at Proupiary produces from the main Saint-Marcet horizon, and three have small outputs. Saint-Marcet is a mushroom structure covered by 1500 m of Flysch. The gas horizons are in a Cenomanian breccia. There is a small output of good oil on the south flank. An 800-km network of pipelines distributes gas. At the end of 1950, 1,160,000,000 m^3 of gas had been produced.

S.N.P.A. has explored the structures of Bastennes-Gaujacq, Garlin, Roquefort, and Pau, finding oil and gas shows but no commercial production. 70,000 m had been drilled at the end of 1950. Lacq was discovered at the end of 1949 on a geophysical anomaly. Oil was found at 640 m in the Campanian. Productivity varies with the nature of the limestone, some wells having potentials of 7000 tonnes/day. Water drive is active. The oil is under-saturated. At present the output is 700 tonnes/day.

S.N.P.L.M. has no production, although numerous wells have found oil and gas shows. At Vaunage there was high pressure gas. Viscous crude has been found in the Oligocene in three wells at Saint Jean de Maruejols. Paraffin-base light oil was found at 1400 m at Coulobres, but three further wells failed to find the lenticular low-porosity Permo-Triassic sandstone. Gabian produced 100 tonnes of oil in 1950, making its total 24,000 tonnes.

Maps show the positions of the concessions, the form of the structures and the gas pipeline network. A section of Saint-Marcet is included. The footage drilled by heavy rigs by the various organizations is shown graphically. Other graphs display the gas and liquid production by years. G. D. H.

Transport and Storage

3080. Trans-mountain pipeline. D. L. Roberts. *Pipe Line News*, July 1952, **24** (7), 23–8. (*Spring Mtng, Amer. Soc. of Mech. Engrs, Seattle, Mar.* 1952.)—Technical and commercial factors involved in the design and construction of the trans-mountain pipeline from Vancouver to Edmonton, scheduled for completion in November 1953. F. M.

3081. Brazilian oil line ready to operate. Anon. *Pipe Line News*, June 1952, **24** (6), 31–2.—A brief account of the crude oil pipeline from Santos to Sao Paulo, including a route and profile map and details of pumping stations. F. M.

3082. Santos Sao Paulo pipeline will aid Brazilian transport. M. da Silva. *World Petrol.*, Aug. 1952, **23** (9), 46, 68.—Railway between Santos and Sao Paulo is overworked and pipeline to carry over 1 million tons pet/year has been constructed. 70% of capital of $8 million pipeline has been invested in the latest types of equipment. Mountain ascent 2379 ft in less than 2½ miles creates a pressure of up to 1020 p.s.i. at the bottom. No pumping station could be provided at the half-way stage because of avalanches. For light oils a 10-inch line was chosen with pressure at seaport end of 900 to 1600 p.s.i. and a 18-inch line for fuel oils at 1020 to 1500 p.s.i. E. B.

3083. Pipelines in France. Anon. *Petroleum*, Oct. 1952, **15** (10), 277.—Briefly describes the natural gas pipelines of southern France, the 10-inch pipeline for transportation of refined products from Le Havre to Paris, and the 16-inch pipeline for crude oil running between the tanker unloading station at Le Havre and the Seine basin refineries. R. E. P.

3084. Trans. Co. pulls New York harbour narrows crossing. J. Klinger. *Pipe Line News*, June 1952, **24** (6), 28–30.—Description of the laying of a 24-inch pipeline across the narrows of New York harbour, which constitutes part of Transcontinental Gas Pipe Line Corpn's 1840-mile system from Texas to New York. The crossing is 6600 ft in length and has a max depth of 100 ft. F. M.

3085. River construction lays dual crossing. Anon. *Pipe Line News*, July 1952, **24** (7), 13–14.—A brief account with illustrations of the dual crossing of the Houston Ship Channel by River Construction Co. A 1700-ft section of ½-inch wall, 20-inch pipe was laid in one push followed by a second crossing using 1700 ft of 16-inch pipe. The crossing is an expansion in the Gas Pipe Line Corpn's system. F. M.

3086. A study of river crossing failures and methods of prevention. J. R. Giese. *Pipe Line News*, June 1952, **24** (6), 21–7. (*API Divn of Transportation, Products Pipe Line Technol. Conf.*)—A discussion of river crossing failures due to river action. The factors involved are described in detail with recommendations for future crossings and maintenance. F. M.

3087. Design for pipeline crossings of alluvial streams. R. T. Knapp. *Oil Gas J.*, 15.9.52, **51** (19), 109.—Careful analyses of stream profiles longitudinal and transverse should be made in the neighbourhood of crossing point.

Flood potentials, turbulent flow, causes of scour, bottom and side scour, resistance to scour, and stream control have all to be considered. G. A. C.

3088. Iraq pipeline. Anon. *Pipe Line News*, Sept. 1952, **24** (9), 21–8.—An account of the planning, construction, and laying of the Iraq Petroleum Co.'s 560-mile, 30-inch pipeline from Kirkuk, North Iraq, to Banias on the Mediterranean north of Tripoli. Two river crossings, the Euphrates and Tigris, were made by use of four 16-inch submarine lines. Pumping stations are powered by diesel engines, and a typical layout is given. F. M.

3089. Analysis and design of unsupported spans. P. E. Reynolds. *World Oil*, May 1952, **134** (6), 216.—The laying of large dia pipe across terrain cut by canyons and gullies introduced the possibility of the unsupported span. This system, apart from the economic advantage, enables the pipe to be kept clear of the effect of floods which would normally scour out any buried lines.

The stresses set up may be classified into transverse and longtitudinal stresses due to the internal pressure, longtitudinal stresses due to temp fluctuation, and longtitudinal bending stresses. The latter are peculiar to this form of pipeline, and have been treated in the same way as those stresses obtained in any uniformly loaded beam. The estimation of the independent stresses is simple, but for the estimation of the effect of a stress at right-angles to the primary stress, on the latter, the following equation is recommended: $x^2 - xy + y^2 = 1$, where x is the primary stress, y is the stress at right angles to the primary. In such a design problem, in which so many factors exist, although under certain conditions secondary stresses appear to cancel out, the worst case must be considered for the more representative estimation of the factor of safety.

Data are presented graphically, with special reference to the 34-inch Topcock–Miltipas natural gas line. A. J. H.

3090. Critical flow of gas in pipe. R. F. Nielsen. *Oil Gas J.*, 8.9.52, **51** (18), 129.—No. 490 in the Engineering Fundamentals series deals with formulæ and graphs for critical flow in gas from reservoirs. G. A. C.

3091. Computing flow conditions in gas lines with the McIlroy analyser. C. L. Barker. *Pipe Line News*, Aug. 1952, **24** (8), 21–2. (*Pacific Coast Gas Assn, Fresno, Calif. Mar.* 1952.)—Brief details are given of the McIlroy analyser, which uses a resistor (known as a Fluistor) such that the voltage drop along it varies in a similar manner to pressure drop in pipes. This enables an electrical analogue to be set up and used for pipe-line computations. F. M.

3092. Automatic block valve controls. F. E. Hangs. *Pipe Line News*, July 1952, **24** (7), 29–35. (*Course in Gas Technol, Texas College of Arts and Industries, Kingsville, May* 1952.)—A brief discussion of the available types of automatic block valves with details of the system chosen and operated by Transcontinental Gas Pipe Line Corpn. F. M.

3093. New electrical pressure instrumentation on oil pipelines. M. J. Dabney. *Pipe Line News*, July 1952, **24** (7), 17–22. (*Amer. Inst. Electr. Engrs, New York, Jan.* 1952.)—Details are given of the development and operation of new-type electrical pressure indicating instruments for the operation of petroleum pipeline pumping stations, as used on the Plantation Pipe Line system. F. M.

3094. Automatic and remote control of electrified pipelines. B. M. Mills. *Pipe Line News*, Aug. 1952, **24** (8), 23–7. (*Amer. Inst. Electr. Engrs, New York, Jan.* 1952.)—Details are given of the characteristics of electrically operated pumping stations and of the use of automatic and remote control. The shell products line from Wood River to Columbus is used as illustration with details of the operation of the Shell remote-control system. F. M.

3095. Centralized control in oil pipeline pump stations. C. B. Lester. *Pipe Line News*, May 1952, **24** (5), 21–7. (*Amer. Inst. Electr. Engrs, New York, Jan.* 1952.)—A description with diagrams of the centralized control system used by Mid-Valley Pipe Line Co. The essentials of control are discussed and applied to pumping stations powered by gas diesels, crude burning diesels, and electric motors. F. M.

3096. Microwaves in operation. C. B. Lester and C. A. Compton. *Pipe Line News*, June 1952, **24** (6), 17–20. (*Conf. of Petrol. Industry Electrical Assoc., Tulsa, Apr.* 1952.)—A general discussion of the features of micro-wave communication system as developed for Mid-Valley Pipe Line Co. F. M.

3097. Use of pigs in hydrostatic tests of large pipelines. M. J. Brinckley. *Pipe Line News*, Sept. 1952, **24** (9), 33–7. (*Ann. Mtng Pacific Coast Gas Assoc., Fresno, Calif., Apr.* 1952.)—Details are given of methods developed by the Southern California Gas Co. for hydrostatic testing of 30-inch pipelines, with an account of the testing of the 30-inch line from Blythe to Los Angeles. F. M.

3098. Bending vibrations of a pipeline containing flowing fluid. G. W. Housner. *J. Appl. Mech.*, 1952, **19**, 205.—An analytical investigation is presented of the free vibrations and of the forced motions due to crosswind which are encountered in the design of pipelines supported above the ground. It is shown that the flow of fluid has no beneficial effect upon the vibrations. The fluid velocity causes a dynamic coupling of simple modes of vibration so that the normal modes are of complex shape with 90° out-of-phase components. The solution is given for free vibrations and for steady state vibrations, and it is shown that large amplitudes may be developed due to resonance effects. At low fluid velocities there is little effect upon the vibration of the pipe, but at a certain high critical velocity the fluid flow causes a dynamic instability. L. G. H.

3099. Automatic odorizing of natural gases. W. Dubis. *Nafta*, 1951, **7**, 267–9.—In view of proposed expansion of natural gas pipelines all over Poland an odorizing substance, Detektol, should be introduced at source and to enable linesmen to discover leaks and to warn the consumers. Two designs for odorizing equipment are shown—one consisting of main reservoir, pump, secondary reservoir, float chamber, and odorizing pipe system operated by differential orifice pressure drop; second similar

but with the pump as a standby only and without float chamber. Formulæ for orifice size are given. M. S.

3100. Plastic pipe tested in corrosive area. G. Miller. *World Oil*, Aug. 1952, **135** (3), 258.—The use of plastic pipe for fluids which are particularly corrosive is recommended. The pipe is corrosion resistant, light, easy to transport and to make-up; it is also resistant to paraffin deposition. Disadvantages include its low pressure application (100 p.s.i.) and the need for protection against external damage from rocks and other debris. A. J. H.

3101. Crude sampler portable and automatic. W. E. Roads. *World Oil*, Sept. 1952, **135** (4), 272.—Where companies are piping several grades of crude oil, it is sometimes necessary to have samples of the various grades. The sampler described provides for the continuous sampling of oil, and is self-contained; it may be installed at any convenient point in the line. The size of sample may be kept small even over a long period of time. A. J. H.

3102. Inter-relation between terminal design, operation, and product deliveries. R. B. Rodgers and S. A. Hallberg. *Pipe Line News*, Aug. 1952, **24** (8), 30–3. (*Products Pipe Line Conf., API, Fort Worth, Apr.* 1952.)—Details are given of methods developed by Standard Oil Co. (Indiana) for calculation of tank sizes in relation to terminal storage requirements. Graphical methods are also presented. F. M.

3103. Factors affecting conservation of products in storage. S. S. Smith and G. D. Harden. *Pipe Line News*, Aug. 1952, **24** (8), 34–6. (*Products Pipe Line Technology Mtng, API, Fort Worth, Apr.* 1952.)—Experimental data are presented on the effect of different types and colours of paint on losses from storage tanks. It is shown that white paint reduces the losses to a greater extent than aluminium paint, and that with an 80,000-brl tank the saving may amount to as much as $1750 per annum. F. M.

3104. Underground storage of natural gas. C. E. Cloud. *Pipe Line News*, Sept. 1952, **24** (9), 29–32.—Discussion of the advantages of underground gas storage and a brief description of the storage developed by the Oklahoma Natural Gas Co. and methods used to calculate the optimum storage needs. Some figures of capital investment and development cost are given. F. M.

3105. Underground LPG storage. R. L. Loofbourow. *World Oil*, June 1952, **134** (7), 261.—A review of factors involved in the selection of a suitable location for LPG storage; primarily the storage should be chosen with regard to the production, transportation, and consumption, and should be in tight rock. The expense of lining storage caverns is generally prohibitive. In case of mined chambers, particularly partitioned chambers, the method of removing rocks should receive careful attention. A. J. H.

3106. Glass fibre protects tank bottoms. C. G. Danielson. *World Oil*, July 1952, **135** (1), 226.—The settling of sludge in tanks is a major problem in countering corrosion. This method of protecting the tank consists in laying a glass fibre base, over which is applied a plastic coating. This improves the mechanical strength of the bottom and takes up any movement of the tank bottom without cracking the coat. The protection is economic and satisfactory. A. J. H.

3107. Aramco adopts plastic tanks for desert use. Anon. *World Petrol.*, Aug. 1952, **23** (9), 40–1.—Steel of the two 1000-gal fuel-carrying tanks and 100-gal water tanks of fifteen trucks has been replaced by Laminac polyester resin reinforced with Fibreglas mat. The tanks are constructed by spraying the mould with the resin filled with Celite and white Calco paste, layering with glass mat, and spraying each layer with catalysed resin. Two tank halves are assembled and reinforced with impregnated glass tape and filled with live steam to cause reaction to take place. Repairs are made by cleaning the rupture and closing with plastic impregnated glass mat. E. B.

3108. How fast do your stores move? J. E. Brewer. *Petrol. Process.*, 1952, **7**, 1133–7.—A description is given of the warehouse recently completed at the Wood River, Ill., refinery of Shell Oil Co. A general layout plan of the entire stores area and one of the main stores building are included, together with several interior photographs. Considerable streamlining in the expediting of orders has been achieved with the aid of such facilities as a pneumatic tube system for paper work, loudspeaker intercommunication system, conveyor belt in the main warehouse, and use wherever possible of palletizing and fork lift trucks. The organization consists of four sections: (*a*) material procurement, maintenance of stocks, and departmental accounting; (*b*) receipt, storage, issuance, delivery, and salvaging of materials; (*c*) inventory; (*d*) priorities group. D. W. F.

3109. Drum handling problems solved with new layout. F. W. Shaw, Jr. *Refiner*, Aug. 1952, **31** (8), 91–4.—A modern drum-filling plant is described and generously illustrated. Drums are transported on roller conveyors, and are handled at the rate of eight per min. A. R. H.

3110. Want lower costs on can filling? Anon. *Petrol. Process.*, 1952, **7**, 964–5.—Review of "The control of lube oil can filling," by B. Mellon and R. Beck, presented before Packaging Institute Mtg 22–24 Oct. 1951, New York City. The use is described of a vernier device developed by Gulf Oil Corpn for precise vol control of lube oil can filling. A survey of the normal filling operation revealed that piston displacement, as set by a cam, was the only variable directly affecting vol delivered. A vernier setting on the cam, calibrated in ° F corresponding to the temp of the oil being filled, ensures precise delivery of the desired vol when corrected to 60° F. D. W. F.

3111. Determination of the heat transfer conditions between bitumen and steel. H. Schmitz. *Bitumen, Teere, Asphalte, Peche*, 1952, **3** (4), 91–5.—Conventional heating procedures for insulated or uninsulated containers for bitumen storage or transport are discussed. Formulæ presented for calculation of val for a steam heating installation give results of limited accuracy. For calculation of the magnitude of the heat transfer the type of flow at the interface is important. An apparatus is described and illustrated in which are determined the heat transfer from bitumen to steel as it occurs at the container wall of a bitumen container and the heat transfer from steel to bitumen as it appears at a heating surface. A steel vessel containing bitumen is established in a state of thermal equilibrium by means of an electrical heating spiral. The current supplied converted to heat units = heat loss at the container surface. Results tabulated for *B*80, where VR = heating tube temp; V_w = ext container wall temp; K = heat conduction in k.cal/m² hr ° C; αSL = heat transfer steel to air in k.cal/m² hr ° C; αBS = heat transfer bitumen to steel in k.cal/m² hr ° C; αSB = heat transfer steel to bitumen in k.cal/m² hr ° C; are calc from the following equations:—

$$(a)\ \alpha SL = \frac{Q}{F(V_{ws} - V_L)}$$

$$(b)\ \alpha SB = \frac{Q}{F(V_{Bw} - V_{Bm})}$$

$$(c)\ \alpha BS = \frac{1}{\frac{F}{Q}(V_{Bm} - V_L) - \frac{1}{\alpha SL} - \Sigma\frac{\sigma}{\lambda}}$$

$$(d)\quad K = \frac{Q}{F(V_{Bm} - V_L)} = \frac{1}{\frac{1}{\alpha BS} + \Sigma\frac{\sigma}{\lambda} + \frac{1}{\alpha SL}}$$

Data are presented in curves and discussed. R. T.

3112. Patents. U.S.P. 2,591,044 (26.10.45; 1.4.52). W. C. Bomhardt and E. H. Sorg, assrs to The Glenn L. Martin Co. An oil storage tank in which joints are sealed

with a sealing strip comprising 70% of 96% vinyl chloride/4% vinyl acetate copolymer plus 30% of a polyester of glycol sebacate.

U.S.P. 2,591,098 (24.7.47; 1.4.52). H. A. Quist, assr to Sun Oil Co. A breathing device for oil storage tanks.

U.S.P. 2,592,072 (26.8.47; 8.4.52). W. C. Sanders. A test pipe for attachment to a crude oil storage tank. R. C. R.

Refinery Operations

Refineries and Auxiliary Refinery Plant

3113. New McElroy–Wilshire gasoline plant is dedicated. Anon. *Refiner*, Aug. 1952, **31** (8), 112.—This plant is processing 5 million cu. ft. of gas daily and has a capacity of 16 million cu. ft., which could be doubled merely by installing additional compressor h.p. A salt section underlying the plant is available for the construction of underground storage. A. R. H.

3114. High pressure wet sandblasting cleans Esso refinery. T. J. Innes and J. L. Nanney. *Oil Gas J.*, 15.9.52, **51** (19), **86**.—Standard Oil Company at Baton Rouge employ a hydro-blast unit in a blast room 30 × 11 × 8 ft high for cleaning tube bundles and other equipment by blast guns with a capacity of 60 g.p.m. at 1500 p.s.i.

One man can clean a tube bundle, pump casing, or other item in half time required by two to four men using older methods.

The effectiveness of the method depends on nature and extent of fouling material. An analysis of the effectiveness with shell-side and tube-side cleaning is given. A table shows comparison of increase in heat exchanger coeffs obtained by several cleaning methods. G. A. C.

3115. Heat-exchanger tube-sheet design—2. Fixed tube-sheets. K. A. Gardner. *J. appl. Mech.*, 1952, **19**, 159.—It is shown that "fixed" tube-sheets may be designed in the same way as tube-sheets for floating-head exchangers by substituting a calculated "equivalent design pressure" for the actual hydrostatic pressure. This "equivalent design pressure" is a function of shell-side and tube-side hydrostatic pressures and of the differential free thermal expansion between the shell and tubes. It is shown that thinner tube-sheets are possible with floating-head exchangers. For the purposes of this paper externally packed floating-head exchangers and fire-tube boilers come within the classification of fixed tube-sheet exchangers. L. G. H.

3116. Throw-away filter medium. F. O'Shaugnessy. *Chem. Engng*, 1952, **59** (6), 169–71.—Addition of neoprene latex to paper pulp gives the resulting filter-paper high strength and chemical resistance. The strength in ten common acids, alkalis, and solvents is quoted. The applications where it is cheaper than filter-cloth are listed. D. H.

3117. Formation of gas bubbles from orifices. II. R. Jackson. *Industr. Chem.*, 1952, **28**, 391–8. Production of small bubbles by buoyant detachment alone necessitates use of porous plates, but unless bubbles protected by surface active agents multiple coalescences occur and bubble size is difficult to control. By using rotating perforated cross, bubble size reduced by liquid shear. This method of sideways deformation is simplest practicable means of producing small uniform bubbles of controlled size. Perforated hollow propellers, rotating perforated plates, and open impellers, rotating rapidly over orifices of small ring spargers, all operate on same principle. Survey of quantitative work in this field given, with 114 references. A. C.

3118. How to cure pulsation problems in centrifugal blowers. O. W. Acheson. *Refiner*, Aug. 1952, **31** (8), 87.—Pulsations are caused by a reduction of the throughput to a point below that at which the max pressure is developed. It can be avoided by selection of a suitable blower, provision of a smaller unit for light load periods, or recirculation of gas when the demand falls below the critical point. A. R. H.

3119. Engineering reference section. M. Van Winkle. *Oil Gas J.*, 15.9.52, **51** (19), 131.—Pt 22 on fans and blowers deals with efficiency, h.p., and pressure. A table gives fan characteristics, and graphs show curves for three-blade arrangements of radial fans and one axial flow type fan. G. A. C.

3120. How to get what you want when you order cooling equipment. D. P. Thornton. *Petrol. Process.*, 1952, **7**, 958–62.—No. 7 in a series on how to order equipment. As an aid to the selection and ordering of cooling towers the types of equipment are briefly reviewed, but relative merits are not considered. The choice of wet-bulb approach, design wet-bulb temp, and design tolerances are discussed. The data required for ordering natural and mechanical draft cooling towers and air-cooled heat exchangers are listed. D. W. F.

3121. Thermal insulation for industrial requirements. Pt VI. R. Thomas. *Refiner*, Aug. 1952, **31** (8), 113–16.—Insulation for sub-atmospheric temps is discussed with special emphasis on properties other than thermal resistance. It must be protected from ice formation inside the insulation by the application of a suitable vapour barrier. Mechanical strength, water resistance, and electrical conductivity are also important factors in many applications. A. R. H.

3122. Laminated thermosetting plastics. F. C. McCamly. *Petrol. Engr*, Aug. 1952, **24** (9), C43–6.—The properties of high mechanical strength, corrosion resistance, and low moisture absorption are advantages for the use of laminated plastics as: (1) insulators for electrolytic corrosion protection for flow lines and processing and storage installations; (2) valve heads, seats, seat inserts, and stems for operating pressures up to 2000 p.s.i.g. with highly corrosive oils and gases; and (3) pump and compressor rings, valve balls, thrust plates, and insulators in geophysical equipment. E. K. J.

3123. Refiner's notebook. J. G. Dobson. *Oil Gas J.*, 15.9.52, **51** (19), 137.—No. 138 on instrumentation and waste disposal deals with blending of acid and alkali water and gives a layout for industrial waste treatment plant. G. A. C.

3124. Patents. U.S.P. 2,590,866 (14.4.48; 1.4.52). J. A. Jost and E. Vogt, assrs to Foster Wheeler Corpn. Heat exchanger.

U.S.P. 2,591,719 (24.7.48; 8.4.52). C. C. Peavy and R. M. Shirk, assrs to Houdry Process Corpn. Catalyst regeneration apparatus.

U.S.P. 2,591,879 (24.4.50; 8.4.52). A. C. Russell. Heat exchanger for heating oil.

U.S.P. 2,592,121 (8.7.48; 8.4.52). J. A. Crowley, assr to Socony-Vacuum Oil Co. Regenerating a moving-bed catalyst at a uniform burning rate. R. C. R.

Distillation

3125. Analytical solution for crowded ends of McCabe–Thiele diagram. R. G. Blick. *Refiner*, Aug. 1952, **31** (8), 105–7.—A solution is presented based on the assumption that the equilibrium line near the ends is straight, and this gives the number of plates slightly in excess of the actual number for a normal equilibrium curve. A. R. H.

3126. Calculation of the number of theoretical plates in distillation columns. H. E. Watson. *Industr. Chem.*, 1952, **28**, 339–40.—Using McCabe and Thiele's method for determining the number of theoretical plates in a column, it is often difficult to draw an accurate equilibrium diagram. Increasing the scale of the diagram does not help, because experimental points on which x (mol % volatile component in liq) and y (ditto in vapour) are based become widely separated. Difficulty overcome by plotting y–x and not y against x, accuracy of plotting then greater than experimental error. Method illustrated for system benzene–carbon tetrachloride, and it is shown that to distil a product containing 80% carbon tetrachloride from a starting mixture containing 30%, thirty-five plates are required at R.F.R. 40. A. C.

3127. Applied distillation. Pt 1. K. E. Coulter. *Refiner*, Aug. 1952, **31** (8), 95–103.—The fundamentals of batch distillation and continuous distillation of binary and multicomponent mixtures are presented. A. R. H.

3128. Patents. U.S.P. 2,591,010 (10.11.50; 1.4.52). J. F. Rollins and M. E. Brooks, assrs to The Lummus Co. Distillation column, including vertical condenser tubes in the upper part of the column through which coolant is passed, a tube sheet beneath for catching condensate, and a condensate-collecting tray beneath the tube sheet.

U.S.P. 2,591,343 (10.8.49; 1.4.52). A. C. Eld, assr to Gulf Oil Corpn. A tray for a fractionating tower has diametrically opposed liquid inlet and downflow means connected by corrugations having a multiplicity of small and closely spaced perforations. R. C. R.

Absorption and Adsorption

3129. Silica gel adsorption for separation of liquid hydrocarbons. E. K. Seybert. *Petrol. Process.*, 1952, **7**, 1150–3.—Attention is given to the choice of the adsorbent in selective adsorption processes, and various characteristics are considered such as particle size as related to optimum percolation rate, average pore size, surface area, packed density, and purity. Being synthetic in origin, the above properties of silica gel can be controlled as desired. Characteristics of some typical grades of adsorbable silica gels are tabulated. Its behaviour as a polar adsorbent is considered. Some of the problems encountered in going from lab scale to large scale operation are discussed.

Several applications of the selective adsorption process in the medical and pharmaceutical fields are reviewed. Brief mention is made of Sun Oil Co.'s continuous process for the selective adsorptive separation of aromatics from petroleum.

Twenty-five references. D. W. F.

3130. Patents. U.S.P. 2,590,872 (25.9.49; 1.4.52). W. W. Kraft, assr to The Lummus Co. Contacting a lub oil with a decolorizing/stabilizing clay absorbent in a closed vessel and externally transferring clay/oil mixture from the bottom of the vessel to the top through an attenuated path into which is injected a heated inert gas, simultaneously scrubbing the mixture in the attenuated path and removing volatiles from the upper part, and clay and treated oil from the lower part, of the vessel.

U.S.P. 2,592,453 (6.3.48; 8.4.52). J. P. Morgan and J. A. Patterson, assrs to Standard Oil Development Co. Separating *cyclo*hexane from normal hexane by counter-current adsorption/desorption using fluidized silica gel, wherein liquid hexane is used to cool hot silica gel and the vaporized hexane is used for transporting the fluidized silica gel. R. C. R.

Solvent Extraction and Dewaxing

3131. Liquid–liquid extraction. R. L. von Berg and H. F. Wiegandt. *Chem. Engng*, 1952, **59** (6), 189–200.—Applications to sulphur removal from gasoline, sulphur dioxide extraction, propane deasphaltizing, and other industrial separations are reviewed. Comparison is made of efficiencies and capacities of spray, packed, baffle-perforated plate, and bubble-cap columns; Koch and York–Scheibel columns; and Podbielniak and Luwesta mixers. The choice of solvents is discussed briefly. D. H.

3132. Patent. U.S.P. 2,592,490 (29.11.49; 8.4.52). J. P. Thompson, assr to Socony-Vacuum Oil Co. Continuous de-waxing of a wax-bearing mineral oil by means of a rotary filter. R. C. R.

Cracking

3133. Ageing of cracking catalysts. P. W. Sherwood. *Erdöl u. Kohle*, 1952, **5**, 488–9.—Factors involved are discussed, distinction is made between cat activity (conversion of feed to product of different b.p.) and selectivity (gasoline yield); the latter can be assessed in terms of coke-factor, the greater the latter the less the selec-

tivity. Ageing can be either normal, causing decrease of activity, or abnormal, resulting in loss of selectivity. Purely thermal effect on normal ageing is not evident till 760° C is exceeded; above 620° C SO_2 and NH_3 cause lowering of selectivity. Natural cat, due to its Fe content, is more susceptible to H_2S than synthetic. Products contributing to cat ageing are listed; these include Na salts, which affect activity without altering selectivity. V. B.

3134. Evolution of a fluid cat cracker. Anon. *Petrol. Process.*, 1952, **7**, 963.—The Orthoflow fluid cat-cracking unit compared in structural size with previous units of similar throughputs is much more compact. A few features of the new design are mentioned. D. W. F.

3135. Alberta's biggest cat cracker opens. H. C. Plummer. *Petrol. Engr*, Aug. 1952, **24** (9), C29–30.—The new McColl-Frontenac refinery at Edmonton covers approximately 160 acres and processes 5500 b.d. crude. The cat cracker processes 2640 b.d. of gas oil, and the carbon deposited on the catalyst when burned off in the regenerator is equivalent to burning 45 tons of coal per day. The plant includes a catalytic polymerization unit producing high octane gasoline from light gases and a coking unit producing gas, cracked gasoline, heavy gas oil, and petroleum coke from reduced crude. The daily production of petroleum coke is approx 60 tons. A catalytic sweetening process is used to remove obnoxious and deleterious substances. E. K. J.

3136. Thermofor catalytic reforming. J. W. Payne, L. P. Evans, E. V. Bergstrum, and V. O. Bowles. *Petrol. Process.*, 1952, **7**, 966–9. (*From report at API Division of Refining, San Francisco*, 12–16 May 1952.)—As the result of operating a 50-b.d. pilot scale Thermofor catalytic reforming plant Socony-Vacuum Oil Co. Inc. have designed a commercial size unit, a flow diagram of which is given. It is of the regenerative moving-bed type employing a single pressure reactor, with no reheat furnaces and with an atmospheric T.C.C.-type kiln. The catalytic section layout is similar to that of the smaller size bucket-elevator type T.C.C. units. The catalyst selected is a synthetic chromia–alumina gel which promoted good desulphurization. Process features of the kiln, reactor, heat exchange and recovery systems, naphtha and recycle heaters, compression, absorption, stabilization and polymer removal from the gasoline product, and dehydration of the recycle gas stream are discussed. Pilot plant data on ten different types of stock are presented, and reforming for aviation gasoline base stocks is briefly reviewed. The economics of the T.C.R. process are compared with thermal reforming and catalytic polymerization for a typical 30,000-b.d. refinery operating on Mid-continent crude. D. W. F.

3137. Thermal and catalytic cracking. F. Schmeling. *Erdöl u. Kohle*, 1952, **5**, 513–15.—Brief review, giving main operating conditions and product characteristics of various processes. Schematic flow diagrams for thermal, Houdry, fluid-bed, thermal reforming, and Platforming processes are included. V. B.

3138. Catalytic cracking in air-lift T.C.C. units. W. W. Hamilton, S. C. Eastwood, A. E. Potas, and E. A. Schraishuhn. *Petrol. Engr*, Aug. 1952, **24** (9), C5–16.—The application of the air-lift T.C.C. process is described for five schemes of refinery operation to show its adaptability. The results of pilot plant operation give good indication of the behaviour on commercial units as shown in the tables presented. The process can also be adjusted for the production of aviation gasoline base stocks. E. K. J.

3139. How charge stock properties affect carbon deposition in catalytic cracking. C. A. Duval and L. S. Holmes. *Refiner*, Aug. 1952, **31** (8), 109–11.—Experimental data are presented on coke formation produced by cracking sixteen different stocks. Coke formation is correlated by the relation wt % carbon $= K\theta^n x$ (coke factor), where K is a constant, θ the residence time, and n a constant depending slightly on charge properties and temp. An alignment chart is presented for determining the coke factor which is calculated from the physical properties of the charge. A. R. H.

3140. Wide range of feed stocks possible with air-lift T.C.C. W. W. Hamilton, S. C. Eastwood, A. E. Potas, and E. A. Schraishuhn. *Refiner*, Aug. 1952, **31** (8), 71–8.—The air-lift T.C.C. unit is briefly described and a flow diagram presented. Wide flexibility is obtained by varying the space velocity, catalyst to oil ratio, reactor temp, catalyst activity, and recycle ratio. Pilot plant and full-scale data are presented showing the effect of charge stock and these major variables on product yield and quality. Motor gasoline yields on a once-through process range from 37·1% for Slaughter gas oil to 46% for Mirando gas oil at the same carbon factor. A. R. H.

3141. Patents. U.S.P. 2,590,980 (21.6.47; 1.4.52). G. F. Kuhn, assr to Sinclair Refining Co. Regenerating chamber for use in the catalytic conversion of hydrocarbons having a series of spaced, inclined plates, an upper inlet for catalyst, a lower outlet for the catalyst, and means for passing air through the chamber.

U.S.P. 2,592,016 (15.11.49; 8.4.52). W. F. Engel, assr to Shell Development Co. Preparing impregnated catalysts in pelleted form containing a high concentration of heavy metal promoter by double impregnation.

U.S.P. 2,592,403 (9.10.48; 8.4.52). L. P. Evans, assr to Socony-Vacuum Oil Co. Preparing hydrocarbon feed stocks containing asphaltic material for catalytic cracking from broad boiling range stock.

U.S.P. 2,592,404 (27.9.47; 8.4.52). L. P. Evans, assr to Socony-Vacuum Oil Co. Catalytic conversion of the oily constituents in asphalt bearing high boiling hydrocarbon fractions to lower boiling products involving sorption of the oily constituents by the catalyst particles followed by conversion of the sorbed oil.

U.S.P. 2,592,479 (12.2.46; 8.4.52). E. L. Sinclair and W. A. Hagerbaumer, assrs to Socony-Vacuum Oil Co. Catalytic contacting tower for conducting two or more conversions of hydrocarbons in the presence of the same moving contact catalyst stream. R. C. R.

Polymerization

3142. Patent. U.S.P. 2,592,428 (4.6.49; 8.4.52). J. D. Kemp and L. F. Brooke, assrs to California Research Corpn. Polymerizing propene with liquid phosphoric acid whereby at least 50% of the propene is converted to liquid polymer in a single stage. R. C. R.

Alkylation

3143. Patents. U.S.P. 2,592,063 (15.12.49; 8.4.52). C. L. Persyn, assr to Tide Water Associated Oil Co. Apparatus for analysing the acid stream from an alkylation process.

U.S.P. 2,591,509 (30.4.51; 1.4.52). G. Calingaert and H. Shapiro, assrs to Ethyl Corpn. Producing alkyl lead compounds by reacting lead with an alkylating agent containing the desired hydrocarbon radical and having a negative radical which reacts with cadmium with an alkyl cadmium compound RCdX, wherein R is alkyl and X is either alkyl or halide. R. C. R.

Isomerization

3144. Cycloversion, poly units improve refiners' marketing position. E. M. Beebe. *Petrol. Engr*, Aug. 1952, **24** (9), C26–7.—A 1000-b.d. Perco cycloversion unit and 100-b.d. catalytic polymerization unit have been installed at the Peppers refinery, Oklahoma City, to raise gasoline quality with min additional operating and construction costs. Results of operation are given. E. K. J.

3145. Patents. U.S.P. 2,591,367 (21.7.45; 1.4.52). S. H. McAllister, assr to Shell Development Co. Producing higher boiling hydrocarbons by first treating hydrocarbon feed containing an α olefin of at least 4 C atoms to shift the double bond towards the centre and thereafter catalytically alkylating an *iso*paraffin with the isomerized olefin.

U.S.P. 2,591,843 (20.10.47; 8.4.52). J. W. Loy, assr to Phillips Petroleum Co. In the catalytic isomerization of hydrocarbons the improvement consisting in introducing the promoting agent at a plurality of points in the direction of flow of the reactants through a porous catalyst bed to maintain substantially the same rate of conversion throughout the whole of the bed. R. C. R.

Chemical and Physical Refining

3146. Patents. U.S.P. 2,591,525 (21.12.48; 1.4.52). W. F. Engel and P. van't Spijker, assrs to Shell Development Co. Desulphurizing a hydrocarbon oil boiling above the gasoline range by adding a lower boiling straight-run naphthenic fraction to reduce the critical temp of the mixture to at least 20° C below operating temp, then contacting the mixture with a sulphactive hydrogenation catalyst at least 20° C above the critical temp and between 375° and 425° C while compressed to a density of at least 0·25 g/cc and then removing the lower boiling fraction.

U.S.P. 2,591,690 (15.11.49; 8.4.52). K. A. Fischer. De-asphaltizing oils by treatment with hexafluoroxylene at 60° to 120° C.

U.S.P. 2,591,946 (31.1.50; 8.4.52). J. H. Krause and T. B. Tom, assrs to Standard Oil Co. Sweetening high boiling petroleum distillates by removing mercaptans by oxidn in the presence of aqueous KOH containing in solution an active copper compound which acts as oxidn catalyst. R. C. R.

Special Processes

3147. Petrochemicals from Canada. D. R. Keck. *Petrol. Process.*, 1952, **7**, 987–9.—A brief general description is given of the chemical plant now being built by Canadian Chemical Co. Ltd., an affiliate of Celanese Corpn of America, at Edmonton, Alberta. Feed material will be liquefied butane and propane and recovered gases from Edmonton refineries. Partial oxidn with air will be employed to provide a variety of chemicals including methanol, acetone, formaldehyde, acetaldehyde, acetic acid for cellulose acetate production, penta-erythritol, propanol, butanol, propylene glycol, dipropylene glycol, and propyl acetate.

Much of the experience gained at the Celanese Bishop, Texas, plant is being used. Viz., three-quarters of the distillation columns will have sieve-plates and all liquid-liquid extraction towers are being standardized on sieve-plates instead of ring and disk towers as previously used. Thermo-syphon reboilers are also replacing previously standard kettle-type reboilers. D. W. F.

3148. Petrochemical processes. G. Kiddoo. *Chem. Engng*, 1952, **59** (9), 149–68.—Production of chemicals from petroleum is summarized in a diagram, and the following processes are detailed: synthesis gas and hydrogen from natural gas; HCN from natural gas and ammonia; chlorination of methane and ethane; acetylene by thermal cracking, ethylene and propylene by therm cracking; polymerization and alkylation processes; alcohols from olefins; ethylene and propylene oxide and glycols; ketones by dehydrogenation of alcohols; glycerol from propylene, aldehydes and alcohols by oxidn; butylenes and butadiene; aromatics by catalytic reforming; and oxidn of xylenes.

In each of these a flow diagram is given, the operating variables, yields, products, and special difficulties are quoted. D. H.

3149. Petroleum plays a growing role in manufacture of aromatics. H. E. Bramston-Cook. *Petrol. Process.*, 1952, **7**, 1145–8. (*New York Section, Amer. Inst. chem. Eng. Mar.* 1952.)—The growth of the aromatic hydrocarbon chemicals industry in the U.S. is reviewed. Up to the second world war there was little demand for aromatics. The sudden demand during the war was largely met by the Hydroforming process which was first in use to raise toluene production. The process is briefly described, together with the steps involved in producing toluene by this method. The xylenes were produced using the same process with a slightly different naphtha cut. During the war the production of benzene and naphthalene was not significantly increased. Whereas post-war demands for toluene and xylene are near normal,

the greatly increased benzene requirements are being met by Hydroforming, and more recently Platforming, which is also briefly described. This has the advantage of being suitable for the conversion of C_6 ring naphthenes by isomerization, as well as C_6 ring naphthenes, to benzene. Other sources of benzene are the Atlantic Refining Co.'s new process for the catalytic reforming of naphthene fractions and the Houdriforming process. The Thermofor catalytic reforming process is also a potential supplier of benzene. Benzene requirements and uses of the principal aromatics as chemical intermediates are mentioned. D. W. F.

3150. How to reduce phenol in vessels from catalytic petroleum processes. J. A. Biehl. *Refiner*, Aug. 1952, **31** (8), 88–90. (*Industrial Wastes Conf., Lafayette, Ind., May* 1952.)—Laboratory data, obtained on a Scheibel column, are presented on the extraction of phenol from waste waters with hydrocarbons. The % phenol removal is increased as the % aromatics in the solvent is increased. 99·9% of the phenols could be extracted from an effluent originally containing 200 p.p.m. phenols. The operation would require five extraction stages with a solvent containing 66% benzole and 34% fluid catalytic cracked gasoline at a solvent to waste ratio of 2 : 1. A. R. H.

3151. Improved process polymerizes olefins for high quality gasoline. G. E. Langlois and J. E. Walkey. *Refiner*, Aug. 1952, **31** (8), 79–83.—Olefins are polymerized by contacting with a phosphoric acid on quartz catalyst to produce a 98 (Fl clear) octane gasoline. A fine mesh quartz is used to reduce the reactor vol and feed quench is applied to control the temp in the reactor.

Polymerization takes place at 300° to 400° F and 200 to 500 p.s.i.g. The catalyst is prepared in the reactor by soaking the quartz in 70 to 85% phosphoric acid and draining. The effect of process variables is presented, reaction rate is increased by increase in temp and pressure. Reaction variables have little effect upon product quality in the operating region. A. R. H.

3152. Dehydration of natural gas and light hydrocarbon liquids. Pt 1. J. M. Campbell and L. T. Laurence. *Refiner*, Aug. 1952, **30** (8), 65–70.—The basic principles of gas dehydration by compression, cooling, adsorption, and absorption are discussed.

157 references. A. R. H.

3153. Acetylene—coming petrochemical building block? V. B. Guthrie. *Petrol. Process.*, 1952, **7**, 977–81.—Reviews briefly the production of acetylene from carbide and natural gas. In cheap power areas the former process is used, but for coal or power equivalent prices of around $6 per ton natural gas can still be economic at quite a high price. Four major methods for producing acetylene by the cracking of saturated hydrocarbons are discussed. The most favoured in the U.S. is the Sachse process. The major U.S. installations for cracking natural gas to acetylene are mentioned.

The economics governing the choice of acetylene as a starting point for many organic chemicals now made from ethylene are presented. A chart showing the many chemicals derivable from both acetylene and ethylene and the preparation route is given. Vinyl chloride, acrylonitrile, and acetaldehyde being the three most important products from acetylene, are discussed in a little more detail with present and possible future production figures. D. W. F.

3154. Patents. U.S.P. 2,590,869 (21.4.45; 1.4.52). W. O. Keeling, assr to Koppers Company Inc. A cyclic process in which a particulate heat-transmission medium is passed downwardly through a heating zone, then upwardly through a conversion zone in which it contacts and heats a hydrocarbon containing gas plus steam and finally through a cooling zone after which it returns to the heating zone, wherein at least a portion of the gaseous mixture from the conversion zone is cooled by indirect heat exchange and then passed counter-current to said medium in the cooling zone, thereby cooling the medium to below 500° F.

U.S.P. 2,591,096 (8.5.50; 1.4.52). J. E. Penick, assr to Socony-Vacuum Oil Co. Recovering HF from an anhydrous mixture of HF and a fluid inert to HF and to acetonitrile by contacting the mixture with an HF/acetonitrile complex selectively to absorb HF from the mixture and removing absorbed HF from the complex.

U.S.P. 2,591,384 (4.12.47; 1.4.52). D. R. Stevens and R. S. Bowman, assrs to Gulf Research & Development Co. Preparing tri*iso*butene and tetra-*iso*butene by reacting *iso*butene and di*iso*butene at 0° to 110° C in presence of a boron trifluoride–ether complex catalyst.

U.S.P. 2,591,498 (13.4.49; 1.4.52). J. L. Betts and A. L. Chaney, assrs to Standard Oil Development Co. Producing lead scavengers by (1) catalytically brominating xylene (Fe catalyst; 0° to 150° F) and (2) contacting the by-product HBr with a 4/5C atom diolefin (−40° to 100° F) and returning the product to the xylene bromination step.

U.S.P. 2,591, 671(17.9.49; 8.4.52). W. E. Catterall, assr to Standard Oil Development Co. Dehydrating aqueous water-miscible alcohols containing 2 to 4 C atoms by extractive distillation using a water-immiscible alcohol containing 4 to 6 C atoms.

U.S.P. 2,591,672 (3.1.49; 8.4.52). W. E. Catterall, assr to Standard Oil Development Co. Simultaneously dehydrating aqueous alcohols containing 2 to 5 C atoms and preparing an alcohol/gasoline blend by gasoline extractive distillation.

U.S.P. 2,591,699 (14.11.49; 8.4.52). H. V. Hess, G. B. Arnold, and M. L. Drabkin, assrs to The Texas Co. Vapour phase treatment of the product containing water, hydrocarbons, and oxygen-containing compounds (alcohols, esters, acids, ketones, aldehydes) obtained by catalytic conversion of CO and H_2 with a basic compound at elevated temp whereby esters are hydrolysed and acids are decarboxylated to provide a gaseous fraction, gas oil, gasoline, and a water phase containing alydehydes and ketones.

U.S.P. 2,591,712 (13.8.47; 8.4.52). C. E. Morrell and N. C. Robertson, assrs to Standard Oil Development Co. Separating two or more saturated aliphatic alcohols having not more than 4 C atoms and closely related b.p.s by extractive distillation in the presence of a hydrocarbon solvent having a b.p. sufficiently high so as not to form an azeotrope.

U.S.P. 2,591,713 (16.9.47; 8.4.52). E. C. Morrell, C. S. Carlson, and P. V. Smith, assrs to Standard Oil Development Co. Separating a lower mol. wt. C_1 to C_4 alkanol from a high mol. wt. oxygenated compound by extractive distillation in the presence of a hydrocarbon solvent.

U.S.P. 2,591,714 (9.10.50; 8.4.52). E. C. Morrell, assr to Standard Oil Development Co. Separating a C_1 to C_4 alkanol from a ketone of closely related b.p. by extractive distillation in the presence of a hydrocarbon solvent.

U.S.P. 2,591,877 (21.9.48; 8.4.52). N. C. Robertson and R. H. Buchanan, assrs to Celanese Corpn of America. Purifying alchols to remove acetals, carbonyls, and oxides by azeotropic distillation employing methyl *cyclo*hexane as the alcohol entraining agent.

U.S.P. 2,592,089 (8.8.49; 8.4.52). P. F. Warner, assr to Phillips Petroleum Co. Producing stable high mol. wt. mercaptans from olefins by first catalyically removing oxygen-containing compounds therefrom and thereafter treating with H_2S in the presence of a mercaptan synthesis catalyst.

U.S.P. 2,592,377 (8.8.46; 8.4.52). F. T. Barr and H. Z. Martin, assrs to Standard Oil Development Co. Manufacture of CO/H_2 mixtures by passing stream upwardly through a dense fluidized bed of finely divided carbonaceous solids in the presence of fluidizable metal oxide particles. R. C. R.

Metering and Control

3155. Electronics control refining unit. L. Walter. *Petrol. Process.*, 1952, **7**, 1126–7. The new SO_2 extraction unit for the refining of kerosine at the Llandarcy refinery of National Oil Refineries Ltd. is believed to be the first petroleum processing unit in the world under complete electronic control. The liquid level, flow, and pressure controllers actuate standard pneumatic diaphragm motor operated control valves. The control signal is a D.C. electric impulse transmitted from the primary measuring element to the control panel and valves via multi-core telephone cable. An electro-pneumatic relay mounted on the control valve head converts the controller

output signal into terms of valve position. Sketches and photographs illustrate a description of the transmitter and control valve with relay in the case of liquid-level measurement and control. D. W. F.

3156. Maintaining electric controllers. L. E. Markle. *Petrol. Process.*, 1952, **7**, 950–5.—Several important points of good maintenance of electric controllers are considered. Trouble symptoms of the normal motor over-load relay and switch are described with necessary action to be taken to prevent failure. D. W. F.

3157. Flow characteristics of a system with a control valve. G. H. Bouman. *Ingenieur*, 5.9.52, **64** (36), O111–13.—After a brief mathematical treatment a simple graphical method is presented whereby the actual control characteristic of a flow system can be derived from the properties of the control valve, the flow resistance of the system, and the pump characteristic. G. F. T. C.

3158. Infra-red analysers for processes. W. A. Patterson. *Chem. Engng*, 1952, **59** (9), 132–6.—The principles of positive filter and negative filter infra-red gas analysers are explained, and three commercial types for continuous process analysis or control are described. Twenty-three typical applications to process control are listed.

The advantages of non-dispersive analysers over spectrometers are greater ruggedness and stability, lower cost, elimination of mathematical treatments for interfering bands, and greater sensitivity and selectivity. D. H.

3159. Patents. U.S.P. 2,590,918 (3.10.45; 1.4.52). W. E. Barnes, assr to Sun Oil Co. Automatic shut-off valve having means for effecting closure thereof when the fluid flow exceeds a pre-determined velocity.

U.S.P. 2,591,060 (9.3.46; 1.4.52). O. L. Garretson, assr to Phillips Petroleum Co. Excess flow control valve comprising a valve seat and a movable valve member upstream thereof and spring-biased away from the valve seat. R. C. R.

PRODUCTS

Chemistry and Physics

3160. Activated alumina : Heat of wetting by hydrocarbons. V. M. Stone. *J. phys. Chem.*, 1952, **56**, 487.—Heat of wetting of activated alumina Grade X F–21 by *n*-heptane, triptane, methyl*cyclo*hexane, and toluene was determined. For the first two the heat of wetting was *ca* 2 to 7 cal and the last two 4·1 to 4·7 cal/g alumina. On adding water to alumina containing adsorbed hydrocarbons the heat relations indicated relatively complete displacement, whereas the addition of excess cyclic hydrocarbon to alumina containing adsorbed aliphatics there was no thermal evidence of displacement. The data given explain the performance of activated alumina in drying natural gas containing heavier hydrocarbons. The method offers a good approach to other problems of preferential adsorption. L. G. H.

3161. Mechanism of nitration of paraffinic and *cyclo*paraffinic hydrocarbons by nitric anhydride. A. I. Titov and N. V. Shchitov. *Doklad. Akad. Nauk. S.S.S.R.*, 1951, **81**, 1085–8; *Rev. Inst. franç. Pétrole*, 1952, **7**, D102–3.—Reaction can occur even below 0° C in an inert solvent. Under these conditions main reaction products are alkyl nitrates (60 to 65%), nitroparaffins (10 to 15%), and carboxylic acids (10 to 15%). Active nitrating agent is NO_3 formed by the dissociation $N_2O_5 \rightleftharpoons NO_2 + NO_3$. NO_2 has an inhibiting action on reaction rate of nitric anhydride with paraffinic chains. A kinetic study of the reaction indicates that, in order to increase yield of nitro derivatives and reduce that of oxidn products, reaction should be carried out at a high temp, with continual addition of N_2O_5. Thus reaction with *cyclo*hexane in boiling CCl_4 yielded 39% nitro*cyclo*hexane and 41% *cyclo*hexyl nitrate. V. B.

3162. Naphthalene hydrocarbons in paraffinic Groznui crude. E. S. Pokrovskaya, *Doklad. Akad. Nauk. S.S.S.R.*, 1951, **81**, 203–7; *Rev. Inst. franç. Pétrole*, 1952, **7**, D102.—Following on work (Nametkin, *ibid.*, 1950, **73**, 715) resulting in identification

of naphthalenes in Maikop kerosine by the picrate method examination was made of the kerosine fraction (d_4^{20} 0·823, aromatic content 18%) from paraffinic Groznui crude. Cuts at 5° C intervals were taken, d and n rose to a peak at fraction with b.p. 85° to 90° C (2·5 mm). Picrates were prepared by method of Cosciug (*cf.* Abs. **1436—1935**). Following hydrocarbons were identified: naphthalene (in 215° to 240° C fraction); β-methylnaphthalene (239° to 243° C); mixture of α- and β-methylnaphthalenes (243° to 250° C); 1 : 6-dimethylnaphthalene (261° to 264° C); 1 : 7-dimethylnaphthalene (264 to 267° C); 1 : 2 : 7-trimethylnaphthalene (280° to 290° C); 1 : 2 : 5 : 6- and 1 : 2 : 4 : 8-tetramethylnaphthalene (in fraction with b.p. 140° to 150° C at 2·5 mm). V. B.

3163. Contribution to the knowledge of some physico-chemical properties of polystyrene. N. Gralén and G. Lagermalm. *J. phys. Chem.*, 1952, **56**, 514.—Determinations of different physico-chemical constants for a fractionated and several unfractionated samples of polystyrene. A method of graphical fractionation is described, and the result in the case of the unfractionated sample, compared with the result from a summation method based on investigation of the individual fractions. A polymolecularity curve is given for the fractionated sample, and from the data obtained conclusions are drawn concerning the degree of branching of different fractions. In general, the fractions with highest mol. wts. contain more linear molecules than other fractions. The investigation of the other samples shows that the wts and shapes of the mols are extremely sensitive to alterations in the polymerization conditions, and also that elevated temp or the presence of catalyst favour the formation of several maxima in the sedimentation diagram. The mol. wts. from the sedimentation and diffusion measurements are compared with those calculated from the visc data. (Authors' abstract.) L. G. H.

3164. Calculation of phase equilibria by analogy methods. A. Cornu. *Bull. Ass. franç. Tech. Pétrole*, 1952 (95), 3–31.—Requirements for phase equilibria calculation are explained, and three methods of performing such calculations on a Consolidated Engineering Corpn electrical computer are described. Time is, however, lost by modifying the data before this can be fed to the machine, and it is preferable to use an analogy machine where such modification is not required. Use of four such machines is described: a French electronic calculator, an R.C.A. calculator, an M.I.T. design, and a Phillips Petroleum Co. machine which has been specially designed for this purpose. This latter is, however, fully automatic, which increases its cost. A design is presented for a non-automatic machine based on a circuit (diagram given) of variable resistances; a problem involving seven unknowns can be solved in 20 min. V. B.

3165. Possible reactions in the geosynthesis of petroleum. E. G. Curphey. *Petroleum*, Nov. 1952, **15** (11), 297–300.—It is suggested that pyridine derivatives, as present in crude oil, are due to the isomerization of pyrrol nuclei, resulting from the reduction of the bis-methyne-5 : 5′-pyrromethyne fragments in chlorophyl under endogenic conditions, and that phytol may afford useful raw material from which petroleum derivatives may be synthesized. The formation of fatty acids from animal and vegetable matter may, under the influence of ionizing radiations and in the endogenic phase, afford the raw material for natural Fischer–Tropsch processes. Certain catalysts of natural origin, similar to those used in petroleum refining, may account for geochemical phenomena leading to the formation of petroleum. R. E. P.

3166. Viscosities of 0·25 to 90% GR-S rubber solutions. A. B. Bestul, H. V. Belcher, F. A. Quinn, Jr., and C. B. Bryant. *J. phys. Chem.*, 1952, **56**, 432.—Solutions of 0·25 to 90% of x-518 GR-S rubber (polymerized at 50°) and x-558 GR-S rubber (polymerized at 5°) in α-methylnaphthalene and a less extensive range of x-518 G R-S rubber in cetane were prepared using an internal mixer with counter-rotating sigma shaped blades. The flow measurements were made using a McKee worker–consistometer for high concn and Bingham viscometers at low concn. The measurements quoted cover a range of rates of shear and several different temp. L. G. H.

3167. Bench scale method for treating waste by activated sludge. R. H. Coe. *Petrol. Process.*, 1952, **7**, 1128–32.—Biological oxidation with activated sludge is one of the methods used for the purification of water containing organic wastes. A description

is given of an improved apparatus and technique for the bench-scale study of this process and the successful application of the equipment in the laboratory treatment of a typical refinery waste.

The effects of feed composition, loading, phenol content, acidity, alkalinity, sulphides, presence of inorganic salts, and operating variables are reported. Sudden phenol loads impaired operation, although the sludge can be acclimatized to higher phenol loads by gradual increases. Lowering pH from 8 to 6 impairs operation, whereas increase of from 8 to 10 does not. Up to 8500 p.p.m. of dissolved inorganic salts can be tolerated by the activated sludge without loss of efficiency. Several of the above factors have differing effects on biochemical oxygen demand, removal of organic chemicals, and removal of phenols. D. W. F.

Analysis and Testing

3168. Automatic Karl Fischer titration. Apparatus using dead-stop principle. H. A. Frediani. *Analyt. Chem.*, 1952, **24**, 1126–8.—A simple and relatively foolproof automatic apparatus for Karl Fischer-type titrations was needed in order to obtain higher precision and greater rapidity in moisture determinations. A novel electrical apparatus has been developed which utilizes the dead-stop principle, automatically adds the reagent, and differentiates between "true" and "false" or fleeting E.P.s. No vacuum tubes are used. Greater precision is possible because of the automatic addition of reagent and electrical timing of the permanence of the end point. High precision is possible even with laboratories unfamiliar with the Karl Fischer technique. Relatively complex samples may be titrated rapidly, and the possible effect of side reactions is minimized. J. S.

3169. NGAA fractional analysis programme. A. J. Miller. *Petrol. Engr*, Aug. 1952, **24** (9), C31–6. (*NGAA Mtng, Houston, May* 1952.)—Programme has surveyed the analysis of liquid hydrocarbon mixtures by low temp distillation at sixty laboratories in the U.S.A. and the mean deviation from the true value averaged for all components ranged from 0·55 to 1·04 mol % for the samples analysed, the greatest deviation occurring in the separation of isomers. E. K. J.

3170. Determination of individual acetylenes in butadiene and in C_4 hydrocarbon fractions. R. F. Robey, B. E. Hudson, Jr., and H. K. Wiese. *Analyt. Chem.*, 1952, **24**, 1080–2.—As little as 0·02% of ethylacetylene (1-butyne) and vinylacetylene (1-buten-3-yne) can be determined separately and quantitatively in mixtures with other C_4 hydrocarbons by a relatively simple procedure. Acetylenes are precipitated as mixed silver acetylides by treatment with alcoholic silver nitrate solution. The liberated nitric acid is titrated as a measure of total acetylenes. The acetylides are treated with aqueous potassium bromide solution to regenerate the mixed acetylenes in concentrated form. Analysis of the concentrate for the individual acetylenes can be carried out bromometrically as well as by physical methods. Volatile mercaptans (thiols), amines, and other low b.p. substances that react with silver nitrate to liberate nitric acid or deplete the acid liberated from silver nitrate by acetylenes must be removed prior to analysis. Acetylene, biacetylene, and methylacetylene interfere with analyses for ethyl- and vinyl-acetylenes, whilst dimethylacetylene, 1 : 2-butadiene, butenes, butanes, and 1 : 3-butadiene have no effect. J. S.

3171. Determination of acetylenes in specification butadiene. Adaptation of rubber reserve method. R. E. Hyzer. *Analyt. Chem.*, 1952, **24**, 1902–3.—Each of the generally used procedures for determining acetylenes in C_4 hydrocarbons has drawbacks when applied to samples on a routine basis by non-technical personnel. A procedure has been developed by laboratory technical personnel which eliminates these drawbacks. A glass sample tube of known vol is filled with the gaseous sample and allowed to come to room temp and pressure. The sample is then flushed by a stream of nitrogen through a 3-ft column of alcoholic silver nitrate in an absorber. The absorber is provided with a fritted thimble at the bottom for sample. The liberated nitric acid is titrated directly with 0·05N-sodium hydroxide to determine the acetylenes present. One determination takes approximately 10 min. J. S.

3172. Analysis of the system methanol–ethylene glycol–water. F. H. Conrad, M. C. Flint, R. H. Meyer, and J. W. Sjoberg. *Analyt. Chem.*, 1952, **24**, 837–40.—In studying the vapour–liquid equilibrium relations of the system methanol–ethylene glycol–water it was necessary to develop a method of quickly analysing a mixture of these three components. Mixtures of known composition were prepared, and the refractive index and density of each were determined and used to plot a triangular diagram. It is possible by obtaining the refractive index and density of any mixture of these three components to ascertain quickly the percentage composition of each component from these two determinations and use of the diagram. Other samples of known composition were tested for density and refractive index. The results from the triangular plot compared favourably with known compositions, and were considered to be of sufficient accuracy for analysis of mixtures of these three components. J. S.

3173. X-ray diffraction patterns of 2 : 4 : 7-trinitro-fluorenone derivatives of aromatic hydrocarbons. L. J. E. Hofer and W. C. Peebles. *Analyt. Chem.*, 1952, **24**, 822–6.—X-ray powder diffraction patterns were taken of different 2 : 4 : 7-trinitrofluorenone derivatives of aromatic hydrocarbons in order to characterize them more fully and to make future identification more certain. The number of reflections corresponding to the interplanar spacing range 3·1 to 3·5 Å is disproportionately and unexpectedly high. Nevertheless, each diffraction pattern is unique, and it is proposed to use the powder pattern for ultimate positive identification. J. S.

3174. Two viscometers for rapid measurements at definite shearing stresses. R. S. Higginbotham and J. J. Benbow. *J. sci. Instrum.*, 1952, **29**, 221–4.—Two modifications of the cone and plate viscometer are described. The first, for measurements at low shearing stresses (10 to 100 dyn/cm²) employs a phosphor-bronze strip to apply torque to the cone. The second, a direct-reading instrument for measurements at relatively high stress (4500 dyn/cm²), uses a small D.C. motor running at constant torque to drive the cone. The range of the first instrument is 1 to 100 poises at 10 dyn/cm², and 10 to 1000 poises at 100 dyn/cm², that of the second is 0·5 to 30 poises. Their accuracy varies according to the visc and anomaly of the fluid under examination, but the average is about $\pm 5\%$, which is adequate for many purposes. The examination of a sample with either instrument, including filling and cleaning, can be completed within 2 min. C. N. T.

3175. Modified method for viscosity measurement of small samples. J. G. Lillard. *Analyt. Chem.*, 1952, **24**, 1042–3.—Modification of a small-scale viscometer recently described has resulted in improved accuracy, extension of temp range, and reduction in operating time requirements. The viscometer is designed for use with sample vols of less than 0·1 ml. In principle, the apparatus is based upon the rate of flow of a drop or slug of liquid between two fixed points in an inclined capillary tube. To obtain a suitable efflux time the angle of inclination of the tube may be varied in accordance with the visc of the sample. J. S.

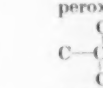
3176. A micro viscometer of improved design. F. M. Luidstone. *Chem. & Ind. (Rev.)*, 6.9.52, 873.—Modifications are made to the micro viscometer described in the *J. Soc. chem. Ind., Lond.*, 21.6.52, p. 189, which gave erroneous results when the temp of the oil differed considerably from that of the room. This defect has been overcome in the modified instruments. No. 1 is for liquids with viscs down to 30 cs and No. 2 for liquids with viscs below 30 cs. In each case the capillary tube is fitted with a stopcock at its upper end so that the column of oil may be held while it is attaining the temp of the bath. The lower end is fused to a wider tube, which fits into a ground glass cone on the short arm of a U-shaped tube, the long arm of which is open to atmosphere above the surface of the bath. Two fine marks are etched on the capillary tube about 10 cm apart. Three drops of oil are required for viscometer No. 1 and eight drops for No. 2, which has a longer effective capillary tube. After charging the viscometer and allowing it to reach the temp of the bath, which is stated to take about 5 min., the stopcock on the top of the capillary is opened and the reading taken between the two marks etched on the tube. Figures quoted, which represent the calibration of two particular instruments, show a repeatability within 0·3%. W. H. A.

3177. Applications for continuous viscosimetry. R. W. Fritzsche. *Petrol. Process.*, 1952, **7**, 1138–43. (*ASME, Cincinnati, Ohio, June* 15–19, 1952.)—The main uses for continuous viscosimetry are classified into five groups. Four types of the more widely employed continuous viscosimeters are described with illustrations. Rotational type—chiefly used for non-Newtonian fluids. Capillary types—flow diagram given of a unit as incorporated in a lube-oil phenol extraction unit. Float type—an adaptation of the rotameter flow meter used in conjunction with a flow regulator. Flow diagrams given for use of this type in two different installations. Plunger type—useful for measuring the higher viscs and automatic control of asphalt penetration. The visc ranges of the float and plunger type are extendable by altering the flow rate of the sample through the instrument.

Temp compensation and sp. gr. correction are mentioned. D. W. F.

3178. Quantitative analysis with infra-red spectrophotometers. Differential analysis. D. Z. Robinson. *Analyt. Chem.*, 1952, **24**, 619–22.—The work described in this paper is a continuation of a programme of evaluating infra-red spectrophotometers for quantitative analysis. It discusses the optimum conditions for quantitative analysis of two-component solutions if both components absorb at the wavelength at which the determination is made. For the most accurate differential analysis the slit widths should be constant from run to run; a single beam curve of the solution should transmit about 40% of the incident energy, and the solvent should absorb as little as possible. In differential analysis with double-beam spectrophotometer the solution is placed in one beam of the spectrophotometer, and a known similar mixture is placed in the other. Increasing the accuracy by changing the solutions in the beams is described. The method can be used to determine small amounts of *cyclo*hexane in benzene. J. S.

3179. Infra-red absorption spectra of tertiary peroxides. A. R. Philpotts and W. Thain. *Analyt. Chem.*, 1952, **24**, 638–43.—It has been suggested that absorptions at 870 and 840 cm^{-1} are characteristic of peroxides and hydroperoxides, respectively. This work sets out to check the validity of these rules and to assess their usefulness in identifying the oxidn products of hydrocarbons in the case of tertiary compounds. The infra-red spectra of *tert*-butyl, *tert*-amyl and phenyl dimethyl methyl peroxides, hydroperoxides, and alcohols are compared. The identification bands are useful for the C_4 and C_5 compounds, but at higher mol. wts. differentiation between alcohol and peroxide becomes difficult. It is probable that these absorptions are due to the



grouping rather that the —O—O-link. When possible, the spectra of the individual components of an oxidn mixture should be recorded so that the compounds can be "fingerprinted." J. S.

3180. Infra-red determination of chlorinated hydrocarbon vapours in air. P. F. Urone and M. L. Druschel. *Analyt. Chem.*, 1952, **24**, 626–30.—Because of the toxicity of chlorinated hydrocarbon vapours in air, their analysis is of importance. Infra-red spectroscopy has a number of advantages in the analysis of compounds of this type. A method has been developed whereby the air is sampled with a cooled scrubbing solvent, and qualitative and quantitative determinations of single or multiple-component vapour–air mixtures are made with an infra-red spectrophotometer using a 1·0-mm cell. A chart of the principal peaks of fifteen chlorinated hydrocarbons is presented, and methods of measuring recorded spectra and preparing standard curves are described. Recoveries averaged 97% with an average deviation of 6%. As little as 0·04 mg of chlorinated hydrocarbon per ml of sampling solution can be analysed. By using the proper solvent, the method may be adapted for the determination of vapours of other organic compounds. J. S.

3181. Ozone deterioration of elastomeric materials. Preliminary results of a study by infra-red spectroscopy. A. R. Allison and I. J. Stanley. *Analyt. Chem.*, 1952, **24**, 630–5.—The work described consists of explorative studies with the ultimate objective of developing an accelerated ozone ageing test for elastomers based primarily on compositional changes rather than on the conventional variation in physical charac-

teristics. Infra-red spectrographic techniques were found to be suited for the reflection of accumulation or depletion of specific structural linkages in polymer mols undergoing ozonization. Purified gum specimens of Hevea, GR-S, nitrile rubber, neoprene, and GR-I were dissolved in chlorinated hydrocarbon solvents such as ethylene dichloride or *o*-dichlorobenzene, and subjected to a stream of ozonized oxygen containing approx 50 p.p.m. of ozone. Infra-red spectrograms of films cast from the treated solutions show progressive intensification of clearly defined absorption bands at 2·9 and 5·8 μ, reflecting the functional groups hydronyl and carbonyl respectively. Methods for estimating these changes quantitatively are described by calibration against reference compounds. A means for following ozone degradation of polymers in terms of specific variations in mol. structure is thus available. Basic information of this nature is potentially useful in the development of an accelerated ozone ageing test correlatable with ageing under conditions of actual service. J. S.

3182. Apparatus for conversion of infra-red spectrometer records to percentage transmission. R. A. G. Carrington and J. G. Reynolds. *J. sci. Instrum.*, 1952, **29**, 197–9.—The measurement and replotting as percentage transmission records of the two traces obtained when using single-beam infra-red recording spectrometers is time-consuming, taking at least as long as the actual production of the two traces by the spectrometer. An apparatus is described which enables these measurements and calculations to be made with considerable economy of time and labour. C. N. T.

3183. Determination of hydrocarbon functional groups by infra-red spectroscopy. S. H. Hastings, A. T. Watson, R. B. Williams, and J. A. Anderson, Jr. *Analyt. Chem.*, 1952, **24**, 612–18.—The use of individual component analysis as a means of characterizing hydrocarbon mixtures is not feasible in the higher boiling ranges because of the large number of isomeric mols, and thus it has become necessary to investigate other methods of characterizing them. One method involves the determination of functional groups, such as methyl and methylene, making up the hydrocarbon mols. The infra-red absorptivities of a number of pure paraffin, naphthene, and aromatic hydrocarbons have been measured under carefully controlled conditions in the 3·0 to 3·5-, 6·5 to 7·5-, and 12·5 to 14·3-μ regions. In general, these absorptivities are related to the concentrations of the various functional groups giving rise to the absorption, *e.g.*, methyl and methylene groups in paraffins at 3·38 and 3·42 μ. This type of hydrocarbon characterization should find increasing application, especially as more complex hydrocarbons become available in a pure state, thus providing the means for more accurate calibration with a min of extrapolation. J. S.

3184. Infra-red spectra in identification of derivative of *cyclo*propane. S. E. Wiberley and S. C. Bunce. *Analyt. Chem.*, 1952, **24**, 623–5.—The presence of a *cyclo*propyl group in an organic mol is difficult to establish by chemical means. The purpose of this work was to establish more definite criteria for the identification of the *cyclo*propyl group by use of infra-red spectroscopy. A study of the infra-red absorption spectra of nine *cyclo*propyl derivatives obtained with a lithium fluoride prism has shown that bands characteristic of the *cyclo*propyl group occur at approx 3·23 and 3·32 μ. The wavelength difference for these two bands is similar to that for *cyclo*propane gas. Use of these two bands for the identification of the *cyclo*propyl group in conjunction with the previously reported bands at 9·9 and 10·9 to 11·7 μ yields more conclusive proof of the existence of the *cyclo*propyl ring system. A study of *cyclo*butyl and *cyclo*pentyl derivatives in the carbon–hydrogen region of 3·4 μ should yield similar selective assignments for these ring systems. J. S.

3185. Ultra-violet spectrophotometric method of determining *p-tert*-butyl catechol. G. G. Campbell and S. A. Tacker. *Analyt. Chem.*, 1952, **24**, 1090–2.—A small quantity of *p-tert*-butyl catechol is added to butadiene under certain conditions to inhibit peroxidation of the monomer and subsequent formation of polymers. An ultra-violet spectrophotometric method of analysis has been developed for the determination of total *p-tert*-butyl catechol in polymerization-grade butadiene. The ultra-violet procedure may be used as an alternative for chemical or colorimetric methods normally used. Briefly it consists of weathering the sample of butadiene to dryness, dissolving the

residue in distilled water, and measuring the light absorption of the aqueous solution at a wavelength of 279 mμ. The *p-tert*-butyl catechol content is proportional to the absorbance. J. S.

3186. Direct spectrophotometric titrations with bromate–bromide solutions. F. B. Sweetser and C. E. Bricker. *Analyt. Chem.*, 1952, **24**, 1107–11.—The use of a standard bromate–bromide solution for analytical determinations has received much attention. Because the E.P. of these titrations is usually determined indirectly or by means of an irreversible indicator, a more satisfactory method is desirable. Direct spectrophotometric titrations with bromate–bromide solutions were investigated, and a simple bromometric procedure for addition and substitution reactions, as well as for some inorganic applications, was developed. A spectrophotometric E.P. utilizing the absorption of the tribromide ion eliminates indicator errors and side reactions. By choosing a suitable wavelength this procedure can be applied with equal accuracy to 0·25N to 0·001N-bromate–bromide solutions. The method can be applied to the determination of olefins, as well as amines and phenols. J. S.

3187. Refractometric adsorption analyses. Use of automatic recording differential refractometer. R. A. Glenn, J. A. Wolfarth, and C. W. De Walt, Jr. *Analyt. Chem.*, 1952, **24**, 1138–43.—The battery-operated continuous automatic recording differential refractometer of Zaukelies and Frost has been adapted to 115-V A.C. operation and investigated as a tool for following the progress of liquid chromatographs of colourless material according to a technique described by Claesson. Integration of the area under the curve in a refractogram obtained through use of the automatic recording refractometer permits quantitative estimation of the individual components in the percolate from an adsorption column by utilizing the additive property of refractive index. The instrument has been used in the quantitative automatic refractometric adsorption analysis of methyl*cyclo*hexane and toluene mixtures. J. S.

3188. A differential refractometer for process control. E. C. Miller, F. W. Crawford, and B. J. Simmons. *Analyt. Chem.*, 1952, **24**, 1087–90.—In present-day petroleum processing plants automatic analyser-controllers are being used to apply the results of continuous analysis directly to process control. A differential refractometer has been developed for such applications, designed to satisfy safety requirements and give reliable indications of changes in refractive index to 0·0001 unit, or better, in outdoor process plant locations. Adequate sensitivity and good stability are achieved without thermostating by using adequate heat exchange pressure equalization between the flowing process and the reference sample. This device may be used to compare the refractive index of two process streams. J. S.

3189. Determination of light hydrocarbons in water. Carbon dioxide stripping method. L. A. Webber and C. E. Burks. *Analyt. Chem.*, 1952, **24**, 1086–7.—In plants processing light hydrocarbons is it desirable to evaluate hyrocarbon losses in process water and steam condensate? The conventional analytical procedures for oil in water fail to recover the light hydrocarbon fractions. An accurate carbon dioxide stripping method has been developed for determining butadiene in water, and may be used also for other light hydrocarbons not recovered by the conventional procedures. The procedure consists of stripping the water sample with gaseous carbon dioxide and collecting the hydrocarbon vapour in a burette over concentrated potassium hydroxide solution. The composition of the gaseous hydrocarbon may be established by Orsat, fractional distillation, or spectrophotometry. J. S.

3190. Routine determination of C_4 hydrocarbons in furfural or absorber oil. R. E. Hyzer. *Analyt. Chem.*, 1952, **24**, 1093–4.—In refinery or butadiene plant operations using an absorber or selective solvent to segregate a C_4 fraction, it is often necessary to determine traces of C_4 hydrocarbons left in lean solvents or absorber oils after the dissolved C_4 hydrocarbons have been distilled out. This analysis can be made with Podbielniak's low-temp Hyd-Robot, but because of the wide differences in b.p.s between the C_4 hydrocarbons and the lowest boiling component in the absorber oil or the solvent it is a somewhat difficult and lengthy operation. A short, vacuum-jacketed, Raschig-ring-packed column equipped with a 500-ml kettle was built, to

which 250 ml of sample plus 50 ml of alcohol as a chaser can be charged. The C_4 hydrocarbons driven overhead are condensed in a graduated pear-shaped centrifuge tube submerged in a dry ice–acetone bath. After vol corrections are applied, this procedure checks the results obtained on the Hyd-Robot very closely and can be run in 15 min as compared with 1 to 2 hr by Hyd-Robot distillation. J. S.

3191. Determination of organic peroxides. Dead stop end point applied to iodometric determination of small amounts of organic peroxides. E. W. Abrahamson and H. Linschitz. *Analyt. Chem.*, 1952, **24**, 1355–6.—A method has been outlined for the quantitative determination of organic peroxides iodometrically using the dead stop E.P. technique. The technique works well in semi-aqueous media, and has several advantages over the visual E.P. technique in that it is applicable in highly coloured solutions or in the presence of insoluble material. It has also been found to function satisfactorily in emulsions if sufficient time is allowed near the E.P. for partition of the iodine into the more aqueous phase. J. S.

3192. Improved ferric chloride test for phenols. S. Soloway and S. H. Wilen. *Analyt. Chem.*, 1952, **24**, 979–83.—The ferric chloride test for phenols as described in standard works on organic analysis fails in many cases. This study shows that this qualitative test can be greatly improved if carried out in organic solvents using a small amount of pyridine as an alkalinizing agent. Some of the best results are obtained in chloroform solution, a medium which had been described previously as giving negative results. All phenols, with some minor exceptions, give the test using these modified procedures. The chemistry of the test is discussed, showing the important part played by the solvent and an added base. J. S.

3193. Determination of sulphur and halogens. Improved quartz tube combustion apparatus. E. D. Peters, G. C. Rounds, and E. J. Agazzi. *Analyt. Chem.*, 1952, **24**, 710–14.—Of the various methods available for the determination of sulphur and halogens in organic materials, the horizontal quartz tube combustion method of Grote is particularly useful, because of its reliability and general applicability. In applying this method to numerous samples originating from research and from manufacture of petroleum products and chemicals, an improved apparatus and procedure have been developed which combine convenience and versatility. The apparatus is of dual unitized design, rugged, and efficient to operate. Satisfactory procedures have been developed for the determination of sulphur and halogen in samples covering a wide range of concentration and volatility. The accuracy of the method in general service work is about 0·03% for either sulphur or halogens. Applicability of the method can be extended to lower concentration ranges by making appropriate changes in apparatus and procedure. J. S.

3194. Tapered-hole disk penetrometer for determining consistency of semi-fluid greases. D. H. Birdsall and B. W. Hotten. *Analyt. Chem.*, 1952, **24**, 892–4.—Semi-fluid greases have consistencies midway between lubricating oils and consistent greases. They are too viscous to be measured by normal viscometric means and yet too fluid to be graded by conventional penetration methods. A variety of methods are now used by manufacturers for grading these products, but there has been no standardization, because no one instrument is satisfactory for all manufacturers and consumers. A tapered-hole disk penetrometer has been designed which provides a satisfactory method of grading semi-fluid greases and is used in conjunction with the ASTM penetrometer. Its use requires no special skill or training other than that required for operation of the ASTM penetrometer. J. S.

3195. Manganous hydroxide method of test for oxygen in butadiene vapours. G. W. Taylor and D. S. Alexander. *Analyt. Chem.*, 1952, **24**, 1083–5.—A comparison of results obtained on determination of oxygen in butadiene vapour by the ASTM manganous hydroxide method of test and by mass spectrometer indicated that the ASTM method gave misleadingly low results. The variables in the test were investigated to determine the cause of the low recoveries. As a result of this investigation the procedure was modified by increasing the volume of 1% manganous chloride

reagent, increasing the amount of shaking of the reaction flask, and incorporating a calibration curve to convert experimental results to actual oxygen values. The modified method gives reliable results for oxygen concentration up to 4% by vol.
J. S.

3196. Ash residues from petroleum catalytic cracking feed stocks. Spectrochemical analysis. E. L. Gunn and J. M. Powers. *Analyt. Chem.*, 1952, **24**, 742–6.—To evaluate the influence of trace metals on the refining, processing, and utilization of petroleum, a sensitive and rapid method requiring a min of effect was developed for measurement of trace of metals in oils in concentrations of 0·1 to 10 p.p.m. The method is employed primarily to inspect catalytic cracking stock for trace metals that may effect catalytic activity. The oil sample is ashed and the residue analysed by a common matrix spectrochemical method. The A.C. arc was found to provide better precision than the D.C. arc method. Three mg of ash are adequate for analysis, and the time for determining nine metals is less than 3 hr. The method permits the determination of silicon, magnesium, calcium, and aluminium, which by other methods would be impractical or impossible to measure in such minute amounts. It is applicable to the routine determination of trace metals in oils other than catalytic feed stocks.
J. S.

3197. Determination of vanadium in fuel oil ash. G. L. Hopps and A. A. Berk. *Analyt. Chem.*, 1952, **24**, 1050–1.—A known quantity, 10 to 20 mg, of the fuel oil ash is weighed into a small beaker, and 2 ml of concentrated H_2SO_4 and 0·5 ml of concentrated HNO_3 are added. The contents of the beaker are carefully heated until all the nitric acid is driven off and fumes of sulphur trioxide are evolved. The fuming is continued until considerable quantities of the salts separate out—usually about 5 min. Cool, add 10 to 15 ml of distilled water and 3 ml of concentrated HNO_3, and heat to dissolve salts. Add 1 ml of 85% phosphoric acid, and 1·5 ml of 0·5 M. sodium tungstate solution. Boil for a few sec; then cool to room temp, transfer to a 25-ml volumetric flask and dilute to vol with distilled water. Measure the transmittance of this solution with a potentiometric-type photoelectric colorimeter, and the amount of vanadium present is determined from a calibration curve. J. S.

3198. Instrument for automatic determination of melting point. R. M. Muller and S. T. Zenchelsky. *Analyt. Chem.*, 1952, **24**, 844–7.—The practical organic chemistry laboratory requires a means for rapidly and precisely obtaining the m.p. of pure organic compounds. An instrument for this purpose is based upon a criterion of melting which is independent of the operator, and it employs samples of micro size. The instrument consists of an electrically heated hot stage with a servo potentiometer temp indicator. A photoelectric sensing and control circuit arrests the servo motor at the m.p. and maintains the temp indication for as long as desired. Heating rates as low as 0·5° C/min are obtainable, and the temp may be read to 0·25° C. M.p. determinations on known compounds made with the instrument after calibration give precision and accuracy well within the limits required by the practical organic chemistry laboratory. The results are in agreement with those obtained by the capillary method.
J. S.

3199. Dielectric indicator for column chromatography. D. E. Laskowski and R. E. Putscher. *Analyt. Chem.*, 1952, **24**, 965–9.—The purpose of this work was to determine the feasibility of using a dielectric constant-sensitive device for detecting colourless components in the effluent stream of a chromatographic column. The instrument chosen for this work was the Thermocap relay. With properly designed dielectric cells this instrument has proved to be a very sensitive detector of small changes in the dielectric properties of the eluate. This method has been used to separate into fractions chromatographic runs made on oxidized Pennsylvanian oils. J. S.

3200. Elimination of errors due to stray capacitances in certain Schering bridge measurements. H. C. Hall. *J. sci. Instrum.*, 1952, **29**, 224–5.—A description is given of a simple method of eliminating the effect of stray capacitances between the test specimen and earth, for a certain class of Schering bridge measurements. The method involves only one simple switching operation, and analysis shows that, using this method, the

test specimen may remain connected to the circuit throughout, so that a usual source of difficulty, the variation in strays upon connexion, is avoided.

With a two-terminal test specimen the method dispenses with the need for a Wagner earth, whether the specimen is screened or not. With a guarded (three-terminal) specimen, the method may still be used with advantage if a small difference in potential between guard and low-voltage electrode is tolerated; this, in current practice, is commonly the case. Though the method described applies particularly to the essentially simple type of Schering bridge used in measurements upon petroleum oils, nevertheless, there are other types in which its use would appear to be advantageous. C. N. T.

3201. A combustion chamber for the semi-micro determination of carbon monoxide and hydrogen. G. T. Rogers. *J. sci. Instrum.*, 1952, **29**, 201.—A method has been described for the determination of CO and H_2, in which the gases are oxidized by a CuO–KOH bead. The present technique involves a simple apparatus for mixtures which do not contain hydrocarbons. The gases are burnt with a known excess of oxygen on a platinum wire in a combustion chamber which is described in detail. The H_2O and CO_2 are removed by the Leighton–Blacet technique. C. N. T.

3202. Laboratory studies of transient elastic waves. S. Kaufmann and W. L. Roever. *Proc. Third World Petrol. Congr.*, 1951, **1**, 537–45.—Using a block of refinery wax as a medium, research has been carried out on the propagation of elastic waves under conditions of interest to the field geologist. Though the problem and the method of its approach are complicated, the outcome of the work indicates that it may be very fruitful.

Each advance made towards the idealized conditions has given results closer to the predicted ones. It is believed that the oscillatory character of the Rayleigh wave is the effect of either a disturbed source (rather than a point source) or of a layered non-homogeneous medium. It is planned to continue this work with other media, both homogeneous and layered. G. S. S.

3203. Simplified differential thermal analysis of mineral clays. J. J. Glogoczowski. *Nafta*, 1951, **7**, 261–4.—Lack of recording instruments prevents Polish scientific centres from making use of the full Differential Thermal Analysis, and the author presents his own simplified method: A steel block is heated in an electric oven at an even rate. This block has two small holes into which go the sample of clay and a quantity of Al_2O_3, each with thermometer. Temps are recorded every 2 min and graphed in such a manner as to obtain $\Delta T vs T(Al_2O_3)$. Results are repeatable closely. Thermometers can be used up to 500° C, but averaging of repeated experiments is advisable. Frequent checks on fixed points (100° and 444·6° C) are suggested. First steps in general use of this method are satisfactory, but with limited experience they need the support of X-ray analyses to prove their value, since a permanent collection of such graphs is necessary for references. M. S.

3204. Here's how to run octane ratings with 20–40 ml samples. R. F. Galloway. *Petrol. Process.*, 1952, **7**, 970–2.—A micro-method for O.N. determinations as developed and used by Magnolia Petroleum Co. is described. The Standard C.F.R. carburettor system with only minor modifications is employed, enabling O.N.s to be obtained on samples of 20 to 40 ml. Details of the equipment required are given. D. W. F.

3205. Draft [German] standard for acid value and saponification value of spark-ignition engine fuels and petroleum spirit. W. Bieler. *Erdöl u. Kohle*, 1952, **5**, 569–70.—DIN 51778 is reproduced; terms "säurewert" and "verseifungswert" are used rather than the corresponding "zahl," as standard is based on units of mg KOH/100 ml, whilst latter term refers to mg KOH/g. Preference for former unit is to avoid fractional values for low-acidity products. 50 ml sample + 50 ml solvent (benzene/alcohol, 2 : 3) is titrated with N/10 alc KOH using alkali blue indicator. For sap val similar quantity of sample and solvent is refluxed (30 min) with 25 ml N/10 alc KOH. Accuracy for both tests is $\pm 10\%$. V. B.

3206. Measuring specific heat of liquids at high temperatures. Small sample apparatus. N. H. Spear. *Analyt. Chem.*, 1952, **24**, 938–41.—A small sample calorimeter using twin radiation tubes was developed in order to measure accurately at high temp specific heats of new heat-transfer liquids currently under development. No available instruments fulfil these requirements. The calorimeter is easily calibrated, has a temp range to over 300° C, and measures with an accuracy better than 5% on samples of approx 35 ml. Specific heat for one liquid is reported, and data are compared with data on another liquid which was available for measurement by conventional methods. The apparatus is unique in its incorporation of modern laboratory techniques, which eliminate previous difficulties in use of the method. J. S.

3207. Hydrocarbon composition of rubber processing oils. S. S. Kurtz and C. C. Martin. *India Rubb. World*, 1952, **126**, 495–9.—In view of increasing developments in the oil-extended synthetic rubber field, the problem of the influence of oil composition upon rubber quality is currently under investigation. Questions of oil classification and nomenclature are dealt with, and an outline is given of the principles involved in mol type analysis, average mol analysis, and carbon type analysis of hydrocarbon oils.

Examples of three equal mol. wt. oils representing the aromatic, naphthenic, and paraffinic classes are cited, together with the properties and analyses of the oils. The experimental details of the Sun Oil Co. procedure are given. A preliminary chromatographic adsorption fractionation into saturates and aromatics is followed by the estimation of *n*-paraffins in the saturates by the urea adduct method, and the determination of density, refractive index, and visc to obtain the ring analysis. For the analysis of the aromatic fraction, it is necessary to measure the same constants, plus the specific dispersion. In the examples quoted, the naphthenic and paraffinic oils have about the same composition in terms of % naphthenes and % aromatics, but the former has, however, more naphthenic rings in both the saturates and aromatic fractions. A major difference is in the ratio of the paraffinic to the naphthenic carbons. A comparison is given of the analytical results obtained by the Sun Oil and the *n–d–M* (van Nes–van Westen) procedures. Agreement for the samples selected is good, but on some extract oils (for which the *n–d–M* method is not recommended by van Nes and van Westen) the two methods give quite different results. The authors conclude that the simple *n–d–M* analysis should be obtained on all rubber processing oils which are being carefully studied. If the ratio of the % aromatic to naphthenic carbons is less than 1·5, and total ring content is less than 75%, the *n–d–M* analysis only is needed, but above these limits (*i.e.*, outside the applicability range of the *n–d–M* method) the present procedure is recommended. If this shows two oils to be similar, they are more likely to behave the same in rubber than if their similarity has been established only by the *n–d–M* method. C. N. T.

3208. Research on the salts of 2-ethyl hexanoic acid. G. Roberts and E. Pipparelli. *Riv. Combust.*, 1952, **6**, 452–61.—Experiments have been carried out on the anti-knock properties of Co, Bi, Ni, Cu, Zn, and Mn salts of 2-ethyl hexanoic acid comparing them with other anti-knock agents. The Co salt of 2-ethyl hexanoic acid is very different from the other salts concerning its behaviour with antagonists (ethylene chloride, butyl mercaptane). The Co salt of 2-ethyl hexanoic acid, similarly to the organo-metallic anti-knock agents, is attacked by chlorine and sulphur; its behaviour resembles that of the organo-metallic compounds (metal atom linked to carbon atom) more than the non-metallic anti-knock agents. (Authors' summary.)

Gas

3209. New perspectives for the European gas industry. C. Padovani. *Riv. Combust.*, 1952, **6**, 443–50.—Different uses of natural gas obtained from northern Italy's deposits are discussed. Use of natural gas for domestic purposes, also problem of methane reforming are examined. When reforming by pyrolysis is carried out under suitable conditions it is possible to obtain on one hand liquid hydrocarbons (aromatics) and on the other non-toxic gas containing a high percentage of hydrogen and with a heat value of 4200 k.cal/m³. (Author's summary.)

3210. Propane and butane in Polish natural gases. A. Stec. *Proc. Main Petrol. Inst.* (*Ministry of Mining—Katowice*), 1951 (16), pp. 18.—From dry natural gas only 1 to

4% of heavier hydrocarbons are obtained, whereas natural gas from crude bearing wells contains over 35% of C_3–C_5. Exploitation by means of cycling (*without* retrogade condensation) is most productive. Once obtained, these gases are separated by adsorption, washing with oil, or compression, followed in each case by fractionation. Propane and butanes have the following uses: (1) as fuel; (2) for polymerization; (3) for deasphalting and solvent refining; (4) chemical raw materials.

Object of this paper is estimation of C_3–C_4 available for industry. In gases C_3-C_4 were estimated by use of Podbielniak's apparatus, in crudes their proportions were established using Gadaskin's apparatus. Both methods are described, and some results given as tables and graphs.

By passing gas over activated carbon the quantity of gasoline in it was determined. All the experiments are described as well as sampling, which was carried out very carefully. Yearly record of analyses was obtained for future use of industry. Max theoretical exploitation of C_3 should reach 44·25% and butanes 75·49% of gas available, but it is in practice approx half that. To attain full efficiency further research into exploitation and recovery is required. M. S.

3211. Improvement of apparatus for extraction of natural gasoline. Z. Wilk. *Nafta*, 1951, **7**, 235–7. (*Convention of Petroleum Industry Rationalizers.*)—Due to small demands for C_3 and C_4 despite efforts to find use for them, only small size equipment for extraction of these gases is required, except where the gas goes into pipelines leading to consumers' burners. Natural gasoline obtained from topping plants contains a large quantity of these gases, and economically working stabilizers which would not involve a great capital expenditure are needed. There is room for inventiveness in the field of gas mensuration. The existing large stabilizing plants and their operators are often inefficient in maintaining a good quantitative balance and heat economy. Full extraction of propane must be borne in mind. M. S.

3212. Some legal problems and regulations concerning exploitation and distribution of natural gases. K. Jahoda. *Nafta*, 1951, **7**, 279–81.—In 1946 all minerals were nationalized. Legislation now covers the aspects of health and safety, as well as efficiency and economy under the " Law for construction of gas carrying pipelines " of 1946. Department of Mines took the advice of the (Polish) Institute of Petroleum in drafting a set of instructions in 1950. M. S.

Engine Fuels

3213. An experimental investigation into pre-ignition in the spark-ignition engine. D. Downs and J. H. Pignéguy. *Proc. Instn Mech. Engrs* (*Auto. Divn*), 1950–51, 125–49.—Pre-ignition and auto-ignition are uncontrolled inflammation of combustible mixture in an engine by a hot surface, and knock and pre-ignition are different phenomena. Two methods of obtaining pre-ignition ratings were developed and are described. One employs a 500-cc single-cyl water-cooled unit with an electrically heated hot-spot to induce pre-ignition, and in the other a single-cyl air-cooled aircraft engine with an air-cooled pre-igniter is used. A number of pure fuels of paraffin, naphthene, aromatic, and alcohol type were rated for pre-ignition tendency using a rating scale with *iso*-octane as 100 and cumene as zero. Typical commercial motor fuels, motor-racing fuels, and water–alcohol injection fluids were also tested, and the relationship between pre-ignition tendency and temp of hot-spot with normal running and with pre-ignition conditions was established. Effects of engine variables and fuel additives were studied, and some information obtained on fundamental chemical processes involved in hot-spot ignition. Twenty-one literature references and nine pages of discussion are included. U. M.

Lubricants

3214. Cities Service open new grease compounding plant at Chicago. Anon. *World Petrol.*, Aug. 1952, **23**, (9) 32–6.—Grease-making equipment and lay-out of 8,375,000-lb plant is described. There are two contactors 100 lb and 1000 lb, and two 1000-lb, one 8000-lb, and one 15,000-lb kettles of stirrer–scraper type. Batch processing was chosen because of multiplicity of products. E. B.

3215. Patents. U.S.P. 2,591,570 (8.4.49; 1.4.52). S. Manilych and L. L. Lambert, assrs to Socony-Vacuum Oil Co. Cutting oil consisting of a mineral lub oil containing about 0·9% active sulphur and 0·1 to 10% of hydroabietyl alcohol to increase drill life.

U.S.P. 2,591,577 (28.4.50; 1.4.52). J. P. McDermott, assr to Standard Oil Development Co. Lub oil containing 0·01 to 2% of an organo-substituted thiophosphoric acid.

U.S.P. 2,591,586 (30.9.50; 1.4.52). A. J. Morway and A. H. Matuszak, assrs to Standard Oil Development Co. High temp grease is prepared by adding 1 to 4 molar proportions of acrolein, acrolein dimer, or mixtures thereof to a quantity of lub oil, thereafter mixing in a 2·5 to 5% aqueous metal hydroxide solution, adding from 1 to 4 molar proportions of a C_{18} to C_{22} fatty acid, and adding metallic hydroxide solution to ensure saponification of the acid, heating the mixture, and finally adding a further quantity of lub oil equal to the starting quantity.

U.S.P. 2,591,630 (28.11.50; 1.4.52). L. W. Sproule and L. F. King, assrs to Standard Oil Development Co. High temp block lub grease of between 125 and 500 S.S.U. visc comprising 3 to 6 parts of lub oil of between 80 and 125 S.S.U. visc, 1 to 6 parts of lub oil of between 2000 and 5000 S.S.U. visc (viscs at 100° F) and 40%, by wt, of soda-base soap.

U.S.P. 2,591,651 (21.11.49; 1.4.52). D. W. Young, assr to Standard Oil Development Co. Stabilized organic compositions, *e.g.*, grease, lub oil, containing a bis (2-hydroxy 5-alkoxy phenyl) alkane anti-oxidant.

U.S.P. 2,592,140 (9.1.46; 8.4.52). M. M. Holben and W. L. Weeks, assrs to Curtiss-Wright Corpn. Engine lubricating system.

U.S.P. 2,592,435 (14.5.49; 8.4.52). A. E. Lacomblé, assr to Shell Development Co. Lub oil containing an oil-soluble aliphatic nitramine for reducing decomposition product deposition.

U.S.P. 2,592,497 (29.10.49; 8.4.52). W. E. Waddey and E. B. Cyphers, assrs to Standard Oil Development Co. Extreme pressure lubricant comprising lub oil base stock plus 2½ to 10% of a 50% oil concentrate of P_2S_5 treated degras. R. C. R.

Bitumen, Asphalt, and Tar

3216. Contribution to the newer development in asphalt road construction. H. Nüssel. *Bitumen, Teere, Asphalte, Peche*, 1952, **3** (4), 84–7; (5), 113–16. (*Lecture, Res. Assocn for road affairs E. V., Berlin*, 29.2.52).—Pt I. In a forthcoming revised DIN 1995 Pt 1, quality of binders, dropping pt, and duct limits are modified, and the former is no longer a testing method. The min bitumen cont for all emulsion types increases from 50 to 55%, and a 60% type is introduced. Only one cut-back bitumen is specified. Problems involved in road surfacing with hot and cut-back bitumen and former practice in Germany and abroad are reviewed. Eff of adhesives in suppressing adverse effects of H_2O is discussed.

Pt II. Cast asphalt with a min excess of bitumen is used in Germany for floors and road surfacing for the heaviest traffic. Special cut-back bitumen emulsions developed in Germany, stable in storage and containing an adhesive, are applied to stone in any weather in the cold mixing process. Comparative test results, reproduced in curves, show the superiority over other mixtures of wet sand mixtures with cut-back bitumen containing an adhesive. In the Zuyder Sea marshes in Holland, where normal procedures are too expensive and natural stone is absent, a mixture of sand 100 parts, lime 2 to 4 parts, and special cut-back bitumen 5 parts, is used for paths and roads; impure sand and salt do not interfere. The " Retread Process " developed in America for road repairs, the use of coloured bitumens, and the necessity for improved testing methods for asphalt masses are discussed. R. T.

3217. Determination of the quality of road tars on the basis of the point of refraction of the soft pitch type distillation residues. E. Fister. *Bitumen, Teere, Asphalte, Peche*, 1952, **3** (4), 96–7.—Conventional testing of road tars in Germany and Switzerland—dist to 350° C and briquet pitch estimation from soft. pt. or anthracene oil determination—is compared with dist in other countries to 300° C. The material system of

pitch and anthracene oil b.p. > 300° C has soft. pt. *ca* 29·5° to 48° C. This bears a simple but secondary relation to the anthracene oil cont, inter-relation of tar constituents or collodiochem conditions are more decisive. The span between point of refraction and soft. pt. or dropping point is a criterion for temp sensitivity of a binder, but the point of refraction itself is informative. The soft. pt. and point of refraction of residues from a series of road tars topped at 300° C by standard procedure (DIN 1995) were determined. The results are tabulated and curves presented relating the point of refraction of the dist residues to anthracene oil cont. The curves show that the influence of type, quantity, and state of the pitch constituents or micelles decreases with increasing anthracene oil cont. The plasticity span, the temp span between the Fraas points of refraction and soft. pt. R. & B. of the road tars of all standard degrees of visc is 29·5° to 37·7° C, mean 33° C. Clearly the eff of a binder for road surfacing is guaranteed most surely when the plasticity range begins as far as possible below the f.p., *i.e.*, if the point of refraction of the dist residue from a tar topped at 300° C lies below 0° C. R. T.

3218. Pitch coking. F. Rosendahl. *Bitumen, Teere, Asphalte, Peche*, 1952, **3** (4), 104–6; (5), 125–8; (6), 155–6. Pt I. The superiority over other cokes of pitch coke used in Germany for electrode manufacture lies in its low volatile and ash cont, its economical val in its replacement of imported petrol coke. In coking coal tar pitch the chief products are coke, pitch-coke oil, and gas. The coking process is discussed with regard to improvements in design and structural materials for ovens, and charging procedure to avoid damage to the walls.

Pt II. Ovens for pitch coking, of which special designs are illustrated and discussed, should not vary widely from conventional coke ovens. Techniques are discussed for preventing damage by the pressure of liq pitch on the oven walls or by pen into the joints. A normal by-product oven operated with dist gases saturated at 80° C with steam and superheated to 250° C, yields 20% pitch oil and *ca* 250 m^3 gas/ton at N.T.P., consisting of H_2 80%, CH_4 15%, C_6H_6 13·0 g/m^3, also naphthalene.

Pt III. Special designs are described and illustrated. One type used in the oil industry for producing petrol coke yields a coke with a volatile cont 0·5 max. A vertical type with three heating zones processes a medium hard pitch, soft. pt. 65° to 70° C, to a hard dense coke (yield 70 to 80%). Mixtures of pitch and pitch coke with low H_2O cont, or suitably homogenized lignite, peat, or sawdust may be processed. This oven may be used for aftertreatment of cokes obtained by other methods to lower the volatile cont.

Patent specification references are given to all three parts. R. T.

3219. Behaviour of coal tar pitch towards physical and chemical influences. Fr. J. Heinrich. *Bitumen, Teere, Asphalte, Peche*, 1952, **3** (5), 124.—New investigations by the tar chemistry section of the French Gas Inst. on the variation of the products obtained from coal tar with various solvents, types of extraction, concn, and temp are reported briefly. The products obtained are extremely sensitive to heat, Na_2CO_3, H_2SO_4, $H_2C_2O_4$, and superheated steam. Results are tabulated.

Thirteen literature references. R. T.

Special Hydrocarbon Products

3220. Solubility factors enhance uses of petroleum sulphonates. Anon. *Petrol. Process.*, 1952, **7**, 1149.—Oil-free, oil-soluble petroleum sulphonates are discussed with reference to their uses. Their extensive use in industry is due to four major functional properties: (1) emulsification and dispersion of liquids; (2) dispersion and wetting of solids; (3) wetting and dispersion of liquid–solid systems; (4) inhibition of rust and corrosion. D. W. F.

3221. Patents. U.S.P. 2,590,923 (17.8.48; 1.4.52). H. S. Bloch and R. C. Wackher, assrs to Universal Oil Products Co. Producing a drying oil by reacting together (1) an olefinic hydrocarbon, (2) an unsaturated polar compound R—Ar—Q, (wherein Ar represents an aromatic radical, R an alkenyl group, and Q is hydrogen or alkyl, alkenyl, carbonylic, or sulphonic acid group), and (3) a halogen atom, in the presence of an HF catalyst and separating a hydrocarbon phase and an HF phase from which

latter is recovered a drying oil having more C atoms per mol than the olefinic starting material.

U.S.P. 2,591,289 (12.10.49; 1.4.52). R. F. Powell and H. M. Stanley. Frothing agent for froth flotation concentration processes consisting of tetra-alkoxy paraffin having 2 to 5 C atom chain and a methoxy, ethoxy, *n*-propoxy, or *iso*propoxy group attached to each terminal C atom.

U.S.P. 2,591,583 (21.1.49; 1.4.52). L. E. Moody and J. W. Hand, assrs to Standard Oil Development Co. A leaded gasoline contains 0·03 to 5% of an alkyl diamine $H_2N-R-NH_2$, wherein R is an alkyl group containing 2 to 5 C atoms.

U.S.P. 2,591,890 (24.11.48; 8.4.52). R. B. Thompson and L. Schmerling, assrs to Universal Oil Products Co. Production of high mol. wt. resinous and plastic materials having an aromatic structure by reacting an alkyl aromatic hydrocarbon with an organic peroxide.

U.S.P. 2,592,204 (15.2.49; 8.4.52). J. F. Socolofsky and R. V. White, assrs to Socony-Vacuum Oil Co. A mineral oil which prevents salt water rusting contains a small amount of a malic ester obtained by treating malic acid with a C_6 to C_8 alkanol and with a long chain (at least C_{12}) alkanol, removing water and treating the resultant mixture with ammonia or an amine in the presence of a hydrocarbon solvent.

U.S.P. 2,592,383 (28.6.49; 8.4.52). L. J. Blatz, assr to Standard Oil Development Co. Preparing heating oil blends by sweetening an oil boiling between 400° and 650° F to remove mercaptans, caustic washing a cracked distillate of similar boiling range, and blending the thus treated oils.

U.S.P. 2,592,466 (23.9.48; 8.4.52). R. W. Pressing, G. P. Lawless, and B. J. Pettibone, assrs to S. C. Johnson & Son Inc. Preparing paste wax compositions by cooling a solution of wax in a solvent whilst in a condition of streamline flow.

R. C. R.

Derived Chemical Products

3222. Petrochemicals and cellulose acetate from plant at Edmonton, Canada. D. R. Keck. *Petrol. Engr*, Aug. 1952, **24** (9), C39–42. (*Chem. Inst. of Canada mtng, Hamilton, Ontario, Mar.* 1952.)—Proposed plant of Canadian Chemical Co., near Edmonton, is described with details of improvements in unit operation design which have been incorporated. Feed stocks will be liquefied butane and propane from gasoline plants and recovered gases from Edmonton refineries. The hydrocarbons are converted to oxygenated chemicals by partial oxidn with air under carefully controlled conditions.

E. K. J.

3223. Catalytic oxidation of naphthalene. P. H. Calderbank. *Industr. Chem.*, 1952, **28**, 291–5.—Vapour phase oxidn of naphthalene to α-naphthaquinone and to phthalic anhydride, using commercial vanadium pentoxide cat investigated over range 310° to 360° C and up to 10% conversion of naphthalene. Rate of oxidn determined by rate of chemisorption of oxygen. Under conditions of these experiments (low temps, low conversions) α-naphthaquinone formed at almost double rate of phthalic anhydride, whereas in commercial practice (high temps, high conversions), latter is formed very much faster. Reason believed to be poisoning of cat on plant scale by other products of naphthaquinone reaction.

A. C.

Miscellaneous Products

3224. New types of synthetic rubber. Anon. *Industr. Chem.*, 1952, **28**, 387–90.—Properties of GR-S and Buna S3 rubbers are compared. By controlling polymerization reaction (particularly by "cold rubber" process), higher mol. wt. rubbers are produced, and these can be used in conjunction with exceptionally high proportions of plasticizing oils. Polymerization of butadiene or butadiene/styrene mixtures by sodium derivatives ("Alfin" catalysis) enables mol. wt. of rubber to reach two or three times that of natural rubber (about 7,000,000) and vulcanizates from this source have remarkably high resilience. World supply and demand position of rubbers is reviewed, and forecast is made that by 1955 there will be an annual deficit of 150,000 tons.

A. C.

3225. Oil absorption tests for pigments. J. R. Bainbridge. *J. Oil Col. Chem. Ass.*, Sept. 1952, **35** (387), 459.—Tests were carried out by Marsden to demonstrate the reproducibility of the B.S.S. method for determining the oil absorption of a pigment. The oil and pigment were mixed together on a plate using a palette knife until a coherent mass was obtained. A new statistical analysis of Marsden's results leads to different conclusions from those drawn by him; and the two sets of conclusions are compared. The following actions are suggested to improve the reproducibility of the process: modification of the procedure to give a more definite E.P., standardization of type of plate used, avoidance of duplicate determinations by the same operator at the same time with the knowledge that he is performing them. D. K.

3226. Radioisotopes in paint technology. G. G. Manov and O. M. Bizzell. *Paint Tech.*, June 1952, **17** (198), 241.—Simple definitions are given of radioisotopes, radiations, and tracers. The production, uses, and restrictions in use of radio-isotopes are discussed. A table is presented showing the properties of some reactor-produced isotopes, *e.g.*, half-life and radiation.

Some possible uses of radio-isotopes in the paint industry include: the radiography of paints, this can be used to reveal the location and size of pinholes and the non-uniformity of a paint coating; transmission and reflection gauges for measuring thickness of paint films; in luminous paints and as tracers.

Tracers can be utilized in the determination of wear resistance, mixing of paints, measurement of the transfer of printing ink and paint, determination of continuity of coating, determination of adsorption of paints and waxes, diffusion studies, metal cleaning, permanency of plasticizers, corrosion studies, determination of the role of driers, activation analysis for determining trace impurities, etc.

Other factors discussed are decontamination of protective coatings and effect of intensive radiation on protective coatings. D. K.

3227. Reaction between styrene and drying oils. A critical survey of published papers. S. Kut. *Paint Tech.*, June 1952, **17** (198), 253. (*Cf.* Abs. 1969, 2770.) Pt IV. The isolation of polystyrene from styrenated linseed oils has been claimed by Bokhout. Powers investigated the reactions between styrene and oleic acid, linseed oil fatty acids and dehydrated castor oil fatty acids; this work and results obtained are discussed. Armitage has reported the reaction of styrene with various oils at 150° C, in the absence of solvent, and in the presence of carbon dioxide. Kappelmeier and co-workers have stressed the need for reliable analytical methods for styrenated products. The work carried out by these investigators is also described. D. K.

3228. Insect control by chemicals. A. W. A. Brown. *Soap, N.Y.*, July 1952, **28** (7), 35.—The subjects of acquired resistance and synergism are surveyed. Attention is drawn to increasing reports of strains of various insects resistant to chlorinated hydrocarbons.

The concept of detoxification is explained. The mechanism of synergism and the possible effect of synergists in inhibiting detoxification are discussed. Work on synergists for D.D.T., pyrethrins, Lindane, and nicotine is reviewed. M. V. J.

3229. Detergents from petroleum. F. D. Snell. *Petrol. Process.*, 1952, **7**, 982–6.—The properties of synthetic detergents are outlined with reference to their mol structure.

Of the many synthetic detergents produced petroleum-based detergents have, since 1948, taken the lead in importance. The principal anionic petroleum-based types are the alkyl aryl sulphonates, the advantageous and disadvantageous properties of which are summarized. With the aid of a batch-process flow sheet the production of this detergent from alkylate is described. The alkylate is a combination of an aryl radical with a petroleum fraction boiling between 345° and 575° F which is sulphonated, neutralized, and then mixed with various builders to improve detergency. Several types of builders in use are mentioned. The sulphonating agent used is 20% oleum. The advantages and disadvantages of the non-ionic detergents are also discussed. These detergents consist mainly of a poly-oxy-ethylene chain for the polar group and an alkyl substituted phenol for the hydrocarbon base. There appears to be increasing interest in non-ionic detergents. D. W. F.

3230. Synthetic detergents—up-to-date. II. Pt I. J. W. McCutcheon. *Soap, N.Y.*, July 1952, **28** (7), 48.—Synthetic detergents are listed by trade name. Manufacturer, chemical composition and % concn, main uses, and physical form are given.
M. V. J.

3231. Evaluation of antibacterial activity. Pt II. E. M. Britt and L. B. Schweiger. *Soap, N.Y.*, July 1952, **28** (7), 138.—Tests undertaken to determine the antibacterial activity of Bactine are described. 1 : 10 dilution of Bactine imparted residual activity to cotton cloths. Toothbrushes were sterilized by immersion for 1 min in Bactine. Surgical instruments contaminated with *M. pyogenes* var. *aureus* were disinfected by 15-min immersion in 1 : 5 Bactine dilution. The counts of *B. subtilis* were reduced markedly. Bactine was shown to have some activity in disinfecting table tops. The antiseptic activity of Bactine was also determined by the Use–Dilution method and the Nungester and Kempf Infection–Prevention method.
M. V. J.

3232. Shampoos as eye irritants. R. A. Cain and R. W. Markland. *Soap, N.Y.*, July 1952, **28** (7), 36.—An account is given of eye irritation studies, using albino rabbits, on commercial shampoos and miscellaneous hair preparations, including a cold permanent wave lotion, neutralizers, rinses, and scalp lotions. Three control solutions were used: (1) soda soap (10 g/100 ml); (2) 40% liq potash soap; (3) dandruff treatment shampoo, reputed to contain 2% quaternary ammonium compound and more than 15% polyoxyethylene non-ionic detergent and known to cause eye irritation.

The soda soap caused least irritation, and the dandruff treatment shampoo most irritation. The commercial shampoos, which included soap, synthetics, and soap synthetic mixtures, caused generally no more irritation than the potash soap, which had a history of safe usage. The miscellaneous hair preparations were generally less irritating than the shampoos. Only the dandruff treatment shampoo caused permanent damage.
M. V. J.

3233. C.M.C.—an aid to washability. J. G. Jarrell and H. B. Trost. *Soap, N.Y.*, July 1952, **28** (7), 40.—Work undertaken to demonstrate the improved soil resistance and washability of C.M.C.-sized cloth is described.

Laboratory tests using cotton test pieces soiled with an aqueous suspension of colloidal carbon showed C.M.C. to be a more effective washing aid when incorporated on the cloth before soiling than when added to the detergent solution in equivalent amounts. Some speculations on the mechanism of this washability feature are offered and supported by tests on other water-soluble film formers, namely, starch, sodium alginate, polyvinyl alcohol, and polyvinyl acetate. C.M.C. was shown to be unique.

Further tests showed C.M.C.-sized cloth to be washed as effectively in a solution of builders only, or even plain water, as unsized cloth in a built detergent solution. (In all tests soil resistance and ease of washing were considered together, *i.e.*, equal depths of soil on sized and unsized fabrics were not obtained.)

Tests utilizing cloth sized with starch, C.M.C., and starch–C.M.C. mixtures over an add-on range of 0·01 to 5·0% (by wt of cloth) showed C.M.C. to have best soil resistance properties with increase in resistance with increased add-on. Washability increased more markedly with increased add-on.

Test with more natural soils also showed C.M.C. to improve washability.
M. V. J.

3234. Inedible fat hydrogenation. Anon. *Soap, N.Y.*, July 1952, **28** (7), 44.—A brief account is given of W. C. Hardesty Co.'s new Canadian plant for production of hydrogenated fatty acids and glycerides for use in the inedible field. M. V. J.

3235. Patents. U.S.P. 2,590,910 (8.5.47; 1.4.52). H. Witcoff and J. R. Roach, assrs to General Mills Inc. Rosin acid esters obtained by reacting pentaerythritol with 1 to 10 mls of glycerol monochlorhydrin to form a polyhydric alcohol and then esterifying substantially all the hydroxyl groups thereof with rosin acid.

U.S.P. 2,590,911 (21.10.48; 1.4.52). H. Witcoff, assr to General Mills Inc. Partial esters comprising a ketone/formaldehyde polyhydroxy condensation product having

at least four hydroxy groups, of which at least one is free and at least one is esterified with a higher fatty acid.

U.S.P. 2,590,971 (24.11.45; 1.4.52). H. L. Johnson and F. J. Bondhus, assrs to Sun Oil Co. Hydrogenated vinylcyclohexene dimer having the empirical formula $C_{16}H_{26}$.

U.S.P. 2,591,239 (5.10.49; 1.4.52). O. C. Dermer and J. J. Hawkins, assrs to Cities Service Research & Development Co. 3-methoxy-4-methoxymethyl tetrahydropyran.

U.S.P. 2,591,245 (11.8.48; 1.4.52). J. D. Edwards, assr to Aluminium Co. of America. Aluminium paste pigment composition comprising 68 to 85% of aluminium flakes, 14 to 28% mineral spirits, and 1 to 4% of stearic and/or palmitic acid.

U.S.P. 2,591,254 (31.12.49; 1.4.52). J. T. Hackmann, assr to Shell Development Co. The production of rubber polymer/sulphur dioxide filaments by spinning the polymer solution in a coagulating bath containing SO_2, wherein the polymer solution contains 0·05 to 5% of a chlorine oxide.

U.S.P. 2,591,539 (3.10.50; 1.4.52). S. O. Greenlee, assr to Devoe & Raynolds Co. Resinous composition comprising 5 to 50% of an epoxide resin (polyether derivative of a polyhydric phenol containing epoxide groups and free functional groups), 25 to 75% of a monobasic acid modified alkyd resin and 10 to 25% of a fusible aldehyde/amine or amide condensate.

U.S.P. 2,591,668 (4.11.47; 8.4.52). A. J. Barry and D. E. Hook, assrs to Dow Chemical Co. Producing phenyl silicon trichloride by reacting benzene with trichlorosilane.

U.S.P. 2,591,710 (6.11.47; 8.4.52). R. F. McCleary and L.W. Devaney, assrs to The Texas Co. Preparing fused cyclic thiophene compounds by reacting a carbocyclic or heterocyclic compound with a saturated alkyl side chain and an adjacent ring H atom with H_2S or volatile thermally labile sulphide in presence of catalyst and at above 700° F.

U.S.P. 2,592,451 (12.1.48; 8.4.52). R. J. Moore and A. Wachter, assrs to Shell Development Co. Vapour phase corrosion inhibitor comprising 2-nitro-thiophene.

U.S.P. 2,592,468 (6.12.47; 8.4.52). W. A. Rex and C. L. Read, assrs to Standard Oil Development Co. Manufacturing cement from oil shale. R. C. R.

Corrosion

3236. What does the management expect from the corrosion engineer? J. K. Peck. *Pipe Line News*, May 1952, **24** (5), 40–2. (*Amer. Gas Assoc. St. Louis, Mo., Oct.* 1951.)—A discussion of the duties and functions of a corrosion engineer in pipeline and gas transmission company. F. M.

3237. Magnesium anode installations in the gas distribution system. H. M. McCamish. *Pipe Line News*, July 1952, **24** (7), 36–8; Aug. 1952, **24** (8), 37–40. (*Paper to Technical Services Conf., Pacific Coast Gas Ass. Calif., Mar.* 1952.)—Details are given of cathodic protection of gas lines by magnesium anode installations. The type of installation and number in relation to the area served are discussed and illustrated. F. M.

3238. Application of hot applied coal tar coatings for pipelines. N. T. Shideler. *Pipe Line News*, June 1952, **24** (6), 38–41.—Details are given of the methods of applying coal tar base coatings, including cleaning the pipe, priming, various methods of application, inspection, patching, and handling. F. M.

3239. Sweet oil well corrosion. H. L. Bilhartz. *World Oil*, Apr. 1952, **134** (5), 208. (*API District Mtng, Shreveport, Mar.* 1952.)—In view of the greater danger of failures in H.P. (high pressure) wells, corrosion of the well tubings has been recognized for some time as a major contributing factor to failure. Recently, however, the occurrence of many failures of L.P. (low pressure) tubings required that a comprehensive investigation be commenced for the general study of sweet oil corrosion. This was directed by

the NACE (National Association of Corrosion Engineers), and the data quoted have been analysed from questionnaires sent out to thirty companies.

Corrosion in the H.P. tubings took the form of general pitting, and was attributed principally to carbon dioxide attack, and secondarily to organic acids which were found in relatively large concentrations. Scale formation was considered to be of significant interest, as it tended to mask the true conditions of the tubing, as determined by caliper surveys. Owing to the restrictions put on the use of nickel steels, the use of thermo-setting plastics for tube lining has been considered.

Failures in the L.P. wells have been due largely to numerous holes in the tubing walls. The conclusions reached in the initial stages of the investigation indicate that: corrosion does not occur below a critical water/oil ratio, but is dependent also on the fluid velocity and total corrodent; gas lift does accelerate corrosion; organic acids, also carbon dioxide, are very active. The more conventional remedial measures are those using inhibitors. A. J. H.

3240. Carbon and graphite. J. P. Oliver. *Chem. Engng*, 1952, **59** (9), 276. **Hastelloy alloy B.** E. D. Weisert. *Chem. Engng*, 1952, **59** (7), 314. **Hastelloy alloy C.** E. D. Weisert. *Chem. Engng*, 1952, **59** (6), 297.—Physical properties and resistance to 150 corrosives. D. H.

Engines and Automotive Equipment

3241. The performance and weight of automobile petrol engines. D. Bastow. *Proc. Inst Mech. Engrs (Auto. Divn)*, 1950–51, 68–81.—The influence on the power output of automobile engines of various factors such as valve arrangement, head design, O.N., and sp. wt. was surveyed, and the results are presented here. It was felt that many of the conclusions drawn from the survey could be presented most effectively by calculating, as a basis of comparison, the primary dimensions and performance characteristics for a series of engines, each intended to develop 100 b.h.p. The series is divided into two groups, one to run on 70 octane and the other on 80 octane. The particulars of these specimen engines are tabulated.

Seven pages of discussion and eleven literature references are included. U. M.

3242. An analytical approach to automobile valve gear design. J. L. H. Bishop. *Proc. Instn Mech. Engrs (Auto. Divn)*, 1950–51, 150–60.—The author presents a general mathematical analysis of the conventional three-arc cam, and introduces new terms "eccentricity" and "instantaneous radius." A similar analysis is made of the new cam profile, and a method is given of correcting the cam profile for high-speed operation. The new general equations can be used to deduce a variable eccentricity ramp, in which the purpose is to take up clearance at variable velocity. The effects of the new cam profile on horsepower and thermal efficiency and the effect of the acceleration characteristic on valve spring design are shown. Reduction in spring wt made possible by elimination of valve spring surge is shown by comparison.

Four literature references and four pages of discussion are included. U. M.

3243. An engine lubricating oil filter. Anon. *Engineer*, 1952, **194**, 360.—A new filter unit for use in lub oil systems of I.C. engines of 60 to 120 b.h.p. is described. The filter operates on a centrifugal principle, the rotary motion of the bowl in which dirt is removed being imparted by reaction of jets through which the oil is pumped. At oil pressure of 30 p.s.i. bowl speed is 3500 r.p.m.; at 70 p.s.i. 6000 r.p.m. Using SAE 30 oil at 140° F, throughput at higher pressure is 95 g.p.h. Removal of bowl, cleaning, and reassembly can be carried out within a few minutes. A. C.

3244. Problems of filtration of motor oils. M. W. Philippe. *Industr. du Pétrole*, Oct. 1952, **20**, 589–94.—Problem of filtration of motor oils is to-day more complex following the greater use of detergent oils. Discussion of types of filters, their installation, and efficiency, is followed by the case for detergent oils. Summing up the limitations of filters, it is pointed out that they do not give 100% efficiency; that they are a supplementary aid for maintaining oil in good condition, but do not do away with the necessity for changing oil. E. H.

3245. Significant contributions of the Diesel Research Laboratory (James Clayton Lecture). C. G. A. Rosen. *Proc. Instn Mech. Engrs (Auto. Divn)*, 1950–51, 7–25.—Work achieved during the past twenty years at Diesel Research Laboratory of Caterpillar Tractor Co. is reviewed. Comparative combustion studies, both visual and those comprising study of combustion chamber deposits, were carried out in order to gain understanding of the combustion process through better knowledge of fundamentals of ignition and mechanism of combustion process. The precombustion chamber process is described in detail; a modified precombustion chamber engine was very successful in the comparative studies on the basis of its ability to maintain uniformity over long periods of operation. Author deals with composition of combustion gases, flame duration, temp distribution, mechanism of ignition, combustion chamber deposits, development of fuel injection equipment, pretiming, and precalibrating. The evolution of the fuel pump, characteristics of several types of check and fuel valves, and fuel pump control of engine torque characteristics are examined, as well as spray characteristics and the flow through the fuel-valve orifice. The author comments on materials for cyl liners and piston rings and the effect of fuel inclusions on cyl wear, and also considers lubrication. It is believed that the development of the precombustion chamber principle of combustion has not yet reached its limit of progress, although excellent performance has already been achieved.

Sixteen literature references are included, and the article is fully illustrated.

U. M.

3246. Generation of gas bubbles at the shrinkage boundaries of built-up crankshafts for diesel engines. S. Amari and E. Ando. *Trans. Inst. Mar. Engrs*, Sept. 1952, **64** (9), 185.—Arising from the observation of minute bubbles at the shrinkage boundaries of the built-up crankshaft of a diesel engine after shop trials, Japanese shipbuilders and crankshaft manufacturers investigated the relationship between the generation of these bubbles and the slip of crank webs on crankshafts. Details of experiments, and data obtained from them, are given. The authors' conclusions are that the bubbles are composed of hydrogen emitted through the surface of steel over long periods; the slipping of webs is due to the presence of oil or grease at the shrink-fit surfaces; the bubbling phenomenon bears no relation to the slipping of crankshafts. A report of the discussion is included.

Four references. T. T.

3247. The Pametrada marine gas turbine. Anon. *Mot. Ship*, Sept. 1952, **33** (390), 225.—Reports a 100-hr duration trial carried out this year on the 3500-b.h.p. Pametrada experimental marine gas turbine. Distillate fuel was used, and an average fuel consumption was reached of 0·505 lb/b.h.p.-hr with a corresponding thermal efficiency of 27·9%. Mean output was 3532 h.p., with inlet temp of 1242° F to the h.p. turbine and 1284° F to the l.p. turbine. Turbine speed was 4650 r.p.m. Modifications made to the gas turbine during 1951 are reported. U. M.

3248. A modified Farnboro electric indicator. W. J. R. Roach and J. G. G. Hempson. *Mot. Ship*, Sept. 1952, **33** (390), 243.—The Farnboro indicator records the time, in terms of crank angle, at which the cyl or other pressure to be determined is balanced by an adjustable and known applied pressure. By its means an accurate indication of cyl pressures in marine oil engines can be obtained. Modifications which have been made to the indicator are described and illustrated, and development work in hand is outlined. U. M.

3249. Compression-operated fuel pumps. Anon. *Mot. Ship*, Sept. 1952, **33** (390), 216.—The Archaouloff compression gas-operated fuel system has been fitted on the main propelling machinery of two cargo liners of the Blue Funnel fleet, and the system has been specified as standard for every two-stroke, single-acting engine building for this fleet—totalling eleven vessels. In the Archaouloff fuel injection system the fuel pump, instead of having a mechanical drive, is operated by either compressed air or, as in the case described here, by gas passing from the engine cyl. Among advantages claimed for the system is the fact that a cleaner and simpler engine can be obtained because the need for the camshaft and cam-operated pumps is obviated. In the case of the "Alcinous" described here, which returned recently from her four-months maiden voyage, the main engines and the three generator engines are all operated on boiler oil, and it is reported that there has been a saving in fuel due to better combustion

and the higher mechanical efficiency of the engine. The gas-operated pumps have worked reliably, and combustion has been greatly improved as evidenced by the clear exhaust, fuel consumption, and lower exhaust temp. U. M.

3250. Some notes on aircraft overhaul and maintenance. C. H. Jackson. *J. Royal Aero. Soc.*, 1952, **56**, 619–38.—Paper was presented to the 1950 Technical Conf. of the International Air Transport Association, and also to the Weybridge and Bristol branches of the Royal Aeronautical Society in 1950–51. Safety and economic aspects of overhaul and maintenance are discussed in the light of commercial aims. The main topic considered is a system of progressive overhaul superimposed on the planned maintenance inspections. Examples are cited of the system operated by B.O.A.C. Probable future trends are outlined. C. N. T.

3251. Patents. U.S.P. 2,590,853 (16.10.45; 1.4.52). C. D. Fulton, assr to General Electric Co. Liquid fuel metering and spraying system including a duplex fuel-injection nozzle having a primary slot to which fuel is supplied at a variable initial pressure and automatic metering means for supplying fuel to a secondary slot at a pre-selected function of said initial pressure.

U.S.P. 2,591,039 (17.9.45; 1.4.52). E. B. Barnes. Oil burning air heater with means for draining spilled oil.

U.S.P. 2,591,421–2 (28.6.46; 1.4.52). R. H. Goddard, assr to The Daniel and Florence Guggenheim Foundation. Combustion chambers for high energy liquid fuel.

U.S.P. 2,591,533 (10.2.47; 1.4.52). W. J. J. Fritz, assr to Iron Fireman Manufg Co. Industrial oil burner for heavy fuel oil.

U.S.P. 2,591,549 (17.5.48; 1.4.52). F. Jonker, assr to Shell Development Co. Rotating continuous combustion type combustion products generator and reaction turbine.

U.S.P. 2,591,619 (24.1.49; 1.4.52). J. T. M. Schlamann, assr to Shell Development Co. Two-stroke cycle internal-combustion engine with slide valve.

U.S.P. 2,591,897 (1.4.44; 8.4.52). V. Weber and R. F. Garner, assrs to Robertshaw–Fulton Controls Co. Safety device for fuel burners.

U.S.P. 2,592,068 (19.7.48; 8.4.52). W. A. Ray, assr to General Controls Co. Oil burner safety control system including a thermo-couple system for insertion into the furnace chamber.

U.S.P. 2,592,132 (6.6.45; 8.4.52). G. B. R. Feilden, A. Simmons, and R. Holl, assrs to Power Jets (Research & Development) Ltd. Fuel supply and control system for the continuous supply of liquid fuel to a spill type atomizing nozzle.

U.S.P. 2,592,367 (13.11.46; 8.4.52). G. B. Wood, assr to American Bosch Corpn. Fuel injection system including two pumps, of which one is a master pump, and common delivery and spill lines.

U.S.P. 2,592,385 (27.5.49; 8.4.52). D. M. Borden and P. T. Nims, assrs to Chrysler Corpn. Automatic control for the fuel metering motor of a combustion system. R. C. R.

Safety Precautions

3252. Fog-foam fire protection at Southeastern stations. Anon. *Pipe Line News*, May 1952, **24** (5), 18–20.—The fog-foam system installed in the pumping stations of the Southeastern Pipe Line Co. is described. The system is complete it itself and independent of the station power, wiring or flow system. It is put into action by heat-actuated devices (H.A.D.'s) which are set to trigger at a given rate of temp rise. In operation the nozzles provide a blanket of foam-and-water fog directed at pumps, manifolds, and strainers, etc. F. M.

3253. Extinguishing of well fires. M. Ptak. *Nafta*, 1951, **7**, 269–74.—The cause of all well fires is a spark in an explosive mixture of gases. Cause of ignition may be due to: (1) carelessness; (2) electrical discharge; (3) mechanical impact. During

rotary drilling in the final stages when the well is being brought in, careful preparations are most required. Low visc mud is required, and blow-out preventers must be frequently checked. Preparatory clearing work in case of fire and actual fire extinguishing are described. Surface and underground approach are possible. (*a*) Fire in a well constantly gushing crude can be extinguished by spraying it with a large quantity of water and jets of water directed at the base of the conflagration; (*b*) in a burning well sporadically gushing crude, flooding with mud or water will extinguish the fire; (*c*) large quantity of burning crude at low pressure can be extinguished by building a wall of sand around it and pushing through its base suction pipes to remove unburned crude and, simultaneously, filling up the area enclosed with stones and sand; (*d*) in case of a fire in a well of large quantity and high pressure production a number of sloping large dia pipes is introduced below the burning orifice to remove crude before it reaches the surface; (*e*) for small quantity of crude and gas burning together, an iron bell-shaped funnel collects all flames together. Such funnel must be placed over the burning well, and then jets of water can be used on the flame; (*f*) small gas producing well on fire can be extinguished by cooling the ground around it with jets of water and then working along the flame from bottom upwards with foam or steam or water; (*g*) to separate the flame from source of gas an explosive charge 20 to 50 kg may be exploded at the base of the flame after the ground has been thoroughly cooled with water. The extinguishing of a high-pressure gas producing well on fire by diverting a part of the burning gases is fully described as an example of another method.
M. S.

3254. Minimizing pump station fire hazards. P. D. Phillips and F. O. Stivers. *Pipe Line News*, July 1952, **24** (7), 43–5. (*Pipeline Symp., API, Chicago, Nov.* 1951.)—A general discussion on fire hazards in pump stations with recommendations for protection.
F. M.

3255. Safety in the transport of liquid hydrocarbons. M. F. Zagdoun. *Industr. du Pétrole*, Oct. 1592, **20**, 595–9.—From a lecture given at the Maison de Chimie, Paris. Risks in transport whether by road, rail, or waterway are of two kinds—toxicity or danger of fire and explosion. Safety regulations are discussed according to the means of transport employed.
E. H.

Miscellaneous

3256. Oil nearly $40 billion industry. Anon. *Refiner*, Aug. 1952, **31** (8), 52–7.—By the end of 1952 the U.S. petroleum industry will have a total gross investment of $40 billion. The increase in investment from 1946 is reviewed.
A. R. H.

3257. Refiners look to Pacific north-west. F. Breese. *Petrol. Process.*, 1952, **7**, 1123–5.—With the anticipated completion by 1954 of the Edmonton to Vancouver pipeline keen interest is being shown in the U.S. Pacific northwest border states. The initial capacity of this pipeline will be 75,000 b.d., with an ultimate capacity of 200,000 b.d. The refining capacity in Vancouver is being increased to 39,500 b.d., which will be nearly sufficient for British Columbia's needs. This will leave 35,000 b.d. of oil to be refined in the area or transported by sea to California for refining and reshipment of the products, gasoline production at present being insufficient to meet local demands. None of the oil companies have disclosed plans for building a refinery, though there appears to be a strong possibility of one of the major companies establishing a new refinery, probably on Puget Sound near Seattle. Standard Oil of California is thought to be the only company that could justify a large refinery in this area.
D. W. F.

3258. Future market for aromatics. Anon. *Petrol. Process.*, 1952, **7**, 1154–60.—As a part of the analysis made by Ebasco Services Inc. of the National Bureau of Mines cost estimate for a 30,000-b.d. synthetic fuel coal hydrogenation plant, a study was made of the markets available for the co-product chemicals at present and as forecast for 1955. This section is reproduced in this article, and includes data on production and uses of benzene, toluene, xylene, phenol, cresol, and cresylic acid. A map included shows the principal market areas for aromatic chemicals. Plots of past and estimated future production trends up to 1955 are given for each of these chemicals in

the U.S. The estimated benzene requirements 1951–55 are given in terms of principal usages for which benzene is required. D. W. F.

3259. The sulphur prospect. Anon. *Petroleum*, Oct. 1952, **15** (10), 278–9.—A review of the estimated production, recovery, and consumption of sulphur in 1955. Estimated production of sulphur in Europe in 1955 is 395,000 tons (native) + 304,000 tons (recuperated). Estimated consumption is 1,500,000 tons. It is concluded that supplies of American sulphur to O.E.E.C. member countries will still be difficult in 1955, although the net European deficit is only a small percentage of the production and consumption of the U.S. It will therefore be necessary that some contribution should be made to recuperation from petroleum and coal sources. R. E. P.

3260. Petroleum refining in France. Anon. *Petroleum*, Oct. 1952, **15** (10), 265–76.—A review describing the progress made in the development of the French refinery industry. By the end of 1952, the expected annual crude capacity of the French refineries will be over 22 million tons. This has made it possible not only to eliminate imports, with the exception of small quantities of special oil products, but also to export refined products. During the period May 1951 to Apr. 1952, the indigenous production of crude oil was 3,030,000 brl (double that for the previous year), *i.e.*, 1·7% of the crude processed. Crude imported from the Middle East represented 90·3% of the total, whilst imports from the Western Hemisphere were only 8·0%. The refineries and their estimated outputs are listed, with brief descriptions of the products and processes at each refinery. R. E. P.

3261. German asphalt, petroleum, and bitumen production—a travel report. A. W. Rick. *Bitumen, Teere, Asphalte, Peche*, 1952, **3** (4), 88–90; **3** (5), 118–23.—Petrol and connected asphalt fields are described in detail under Hanover, Hamburg-Holstein, and Emsland areas. Occurrence, characteristics of materials, recovery, processing, products, and interested companies are discussed. Numerous photographs are presented. R. T.

3262. Profits from dynamic balancing. R. W. Lortz. *Petrol. Process.*, 1952, **7**, 956–7.—The possible saving on replacement and repair costs of high-speed rotating machinery by the use of a dynamic balancing machine is said to be very considerable. The machine recently installed by Phillips Petroleum Co. at its Sweeny, Texas, refinery is quoted as an example, the investment in which it is believed will be amortized in eight months' time. The machine and method of operation are very briefly described. D. W. F.

3263. 50 years of petroleum chromatography. H. Weil. *Petrol. Process.*, 1952, **7**, 812–14, 973–6.—Pt I considers empirical and scientific eras. After defining the scope of chromatography a historical review of the discovery and application of this particular method of separation is given. Three eras of chromatography are distinguished, each characterized by a different approach to the chromatographic principle, the first two, the empirical and scientific, being covered in Pt I. The former period is that in which practical use is made of chromatographic separation in a traditional way. During the latter period the principle was evolved as a scientific process, it being applied to the partial fractionation of hydrocarbon mixtures and animal and vegetable fats and oils. This period is further broken down chronologically into four such periods 1900–1914–1934–1945–1947.

Fifty-one references.

Pt 2 is on the technological era. Of the two different methods of isolation of fractions in chromatography, only the one employing flowing chromatography has been used in large scale separations, and so the discussion is restricted to this method. In passing, the Hypersorption and Arosorb dynamic selective adsorption processes are mentioned. Earlier uses of silica gel in the petroleum industry for general refining purposes are discussed.

1947 is taken as being the commencement of a new era of chromatographic technology, as during this year four examples of large-scale application of the technique became known. The pertinent literature on these is reviewed, and one of them, employing radial flow separation, is considered further. The advantages possessed by radial flow over vertical flow systems are presented. Neither method is continuous in operation. A recently developed "automatic" mechanical method for

isolation of the chromatogram zones, an attempt to overcome the batch-wise operation of previous methods, is discussed.

Twenty-eight references. D. W. F.

3264. Petroleum Rationalization Congress in Jedicze and Krosno. Anon. *Nafta*, 1951, **7**, 240–51.—A report on papers read and discussions that followed. M. S.

3265. Some examples of the statistical approach to the planning and interpretation of industrial experiments. B. Saunders. *J. Oil Col. Chem. Ass.*, Sept. 1952, **35** (387), 448.—The use of the Latin Square for studying the effects of certain factors in the wrinkling of an enamel is described. The vehicle was a mixture of alkyd resin and an oleo-resinous varnish. Factors studied were various proportions of alkyd resin, varying amounts of an anti-skinning agent, and the effect of different drier combinations.

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3266. Statistical treatment of experimental data. A. Lord. *J. Oil Col. Chem. Ass.*, Sept. 1952, **35** (387), 437.—Two statistical methods of treating experimental results are discussed. These are the fitting of curved regression lines and the analysis of variance. D. K.

3267. Emulsifying and homogenizing. B. Waeser. *Bitumen, Teere, Asphalte, Peche*, 1952, **3** (4), 98–103.—The theory of emulsion formation, the main principles of construction, and the development of the relevant machines are treated briefly. Structural forms of emulsifiers and homogenizers are described and illustrated. Abundant literature references appear in the text. R. T.

3268. Ground-water contamination by gasoline. J. Müller. *Gas-u. Wasserfach*, 1952, **93**, 205; *Erdöl u. Kohle*, 1952, **5**, 505.—Account of spring contamination by leaks from gasoline storage. Effect of gasoline was to decrease nitrate content of water, although *in vitro* reaction between gasoline and aq nitrate solutions could not be observed; possibly reaction was accelerated by contact with other materials or biochemically. Gasoline could be organoleptically detected in water at concn of 1 in 10^6; such traces can be removed by filtering through activated charcoal. Due to action on surrounding limestone of CO_2, formed by gasoline oxidn, alkalinity and hardness of water were increased. V. B.

3269. Post-war public-service vehicle maintenance. T. H. Parkinson. *Proc. Instn Mech. Engrs (Auto. Divn)*, 1950–51, 105–23.—Pre-war public-service vehicle maintenance schemes are first outlined, and the re-organization of maintenance and aspects of development of vehicle design affecting maintenance technique are reviewed. Trend of component failure during pre- and post-war periods suggests necessity for modification in maintenance practice. Future trends in chassis design are indicated.

Thirteen pages of discussion are included. U. M.

3270. Use of wire-resistance strain-gauges in automobile engineering research, with particular reference to the measurement of strain in vehicle structures. J. R. Bristow, P. Metcalf, and C. H. G. Mills. *Proc. Instn Mech. Engrs (Auto. Divn)*, 1950–51, 27–47.—The properties and methods of using wire-resistance strain gauges, together with applications and limitations, are briefly described, and factors affecting their accuracy and stability enumerated. Determination of static and of dynamic strain are illustrated by reference to measurements made on public service vehicles and typical strain records given. Portable four-channel dynamic-strain recording equipment developed at M.I.R.A. is described.

The article is illustrated and contains thirteen literature references; ten pages of discussions are included. U. M.

3271. Partnership (Address by the Chairman of the Automobile Divn). E. A. Evans. *Proc. Instn Mech. Engrs (Auto. Divn)*, 1950–51, 1–6.—Review of the work of the chemist in relation to that of the automobile engineer. Particular attention paid to development of engine fuels and lubricants, additives, etc. U. M.

ADDITIONS TO THE LIBRARY

Dictionary of Fire Technology. A. S. Minton (editor). London : Chantry Publications Ltd., 1952. Pp. 97.

Prepared by the Institute of Fire Engineers this dictionary of some 10,000 to 15,000 words includes terms of use to the fire engineer. These terms are drawn from building construction, chemistry, fuel technology and combustion, all branches of engineering, metallurgy, petroleum, radio and telephony, simple physics and mathematics, and refractories, with some toxicology and physiological subjects.

Selection and Use of Engine Antifreezes. ASTM Committee D-15. Philadelphia, Pa : ASTM, 1952. Pp. 15. 40c.

Designed to provide practical information to consumers of engine antifreezes and corrosion inhibitors, together with cleaning compounds and stop-leaks.

Australian Scientific Societies and Professional Associations. G. J. Wylie and Nona F. Lowe. Melbourne : Commonwealth Scientific and Industrial Research Organization, 1951. Pp. 142.

A useful collection of information relating to 212 societies and associations in the Commonwealth, divided under thirteen main headings and very adequately indexed. The index also lists publications of the various bodies. For each body information is given as to address and officers, purpose, history, membership and subscriptions, meetings, publications, and other useful information.

General Discussion in Heat Transfer. London : Institution of Mechanical Engineers, 1952. Pp. 496 + xiii. 45*s*.

In September 1951 a general discussion on heat transfer, organized by the Institution of Mechanical Engineers and the American Society of Mechanical Engineers, was held in London and continued at Atlantic City in November of that year.

In all ninety-three papers were presented in five groups, and these, with the discussions at both meetings, are reproduced in this volume.

ASTM/IP Petroleum Measurement Tables. (British Edn). London : Institution of Petroleum, 1952. Pp. 432. 50*s*.

A Comparison of the Extinguishing Effects of Chlorobromomethane, Methyl Iodide, and Carbon Tetrachloride. E. H. Coleman. London : Fire Protection Association, 1952. Pp. 12.

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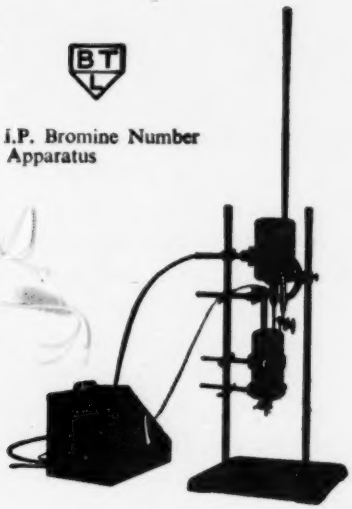

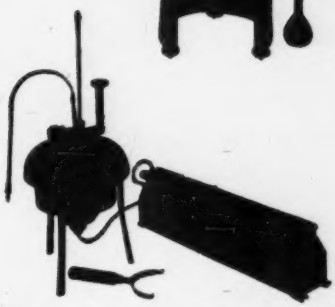

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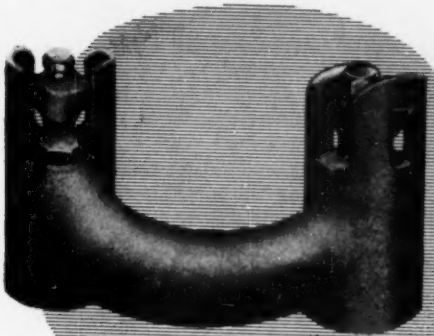

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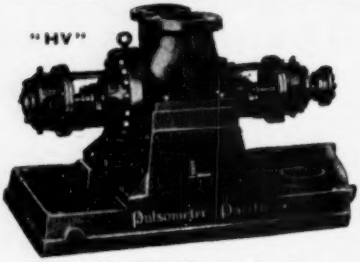

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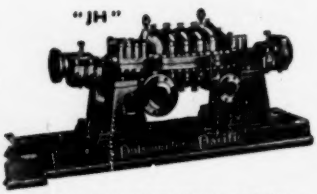

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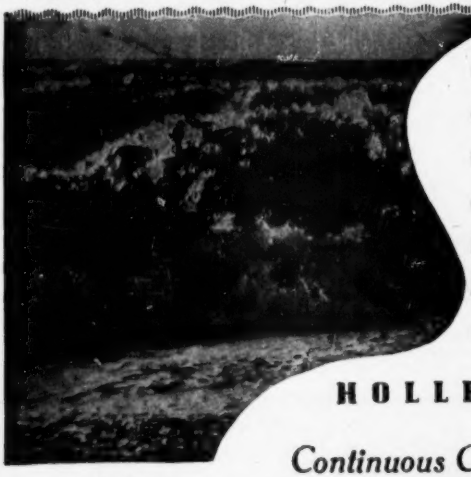

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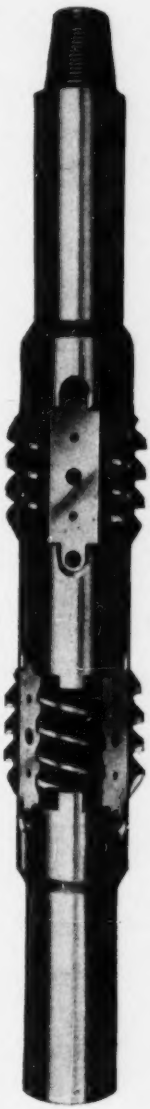

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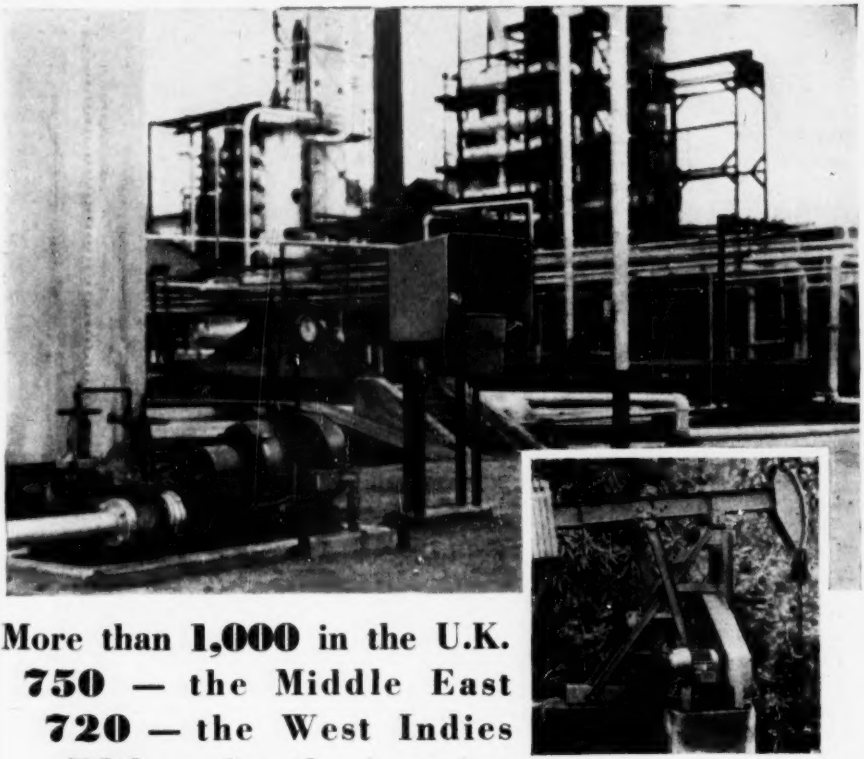

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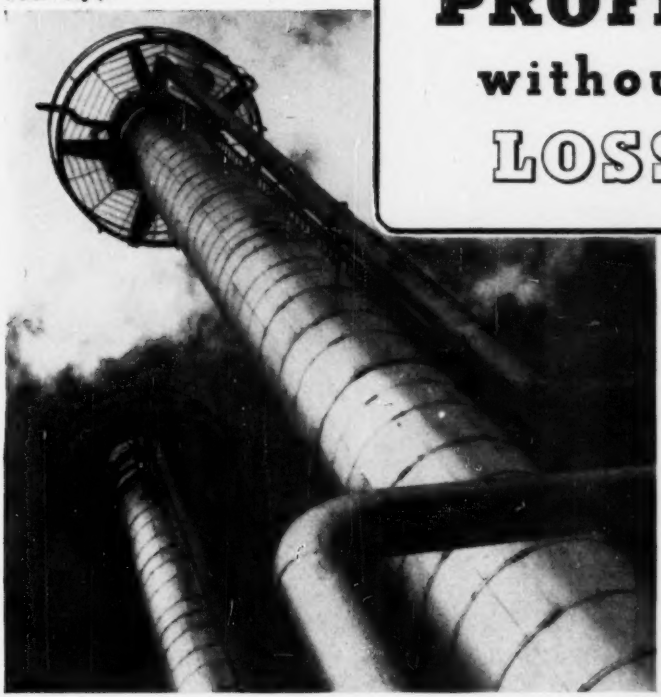

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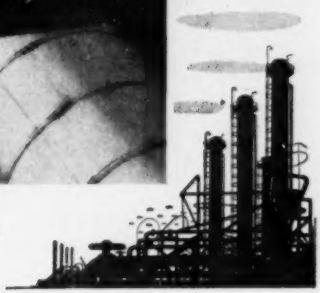

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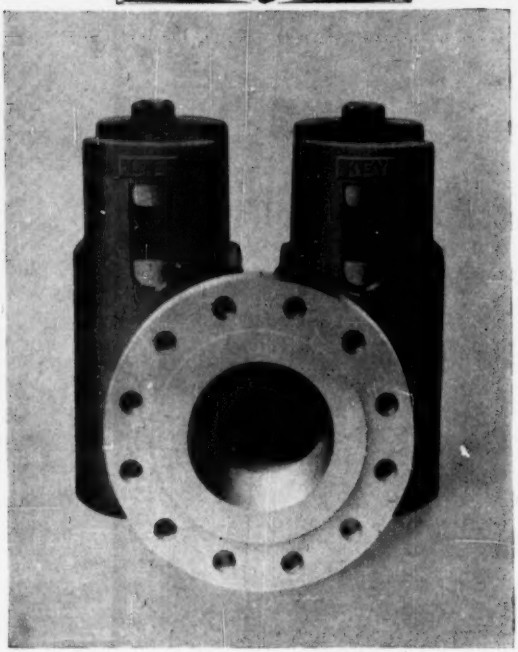

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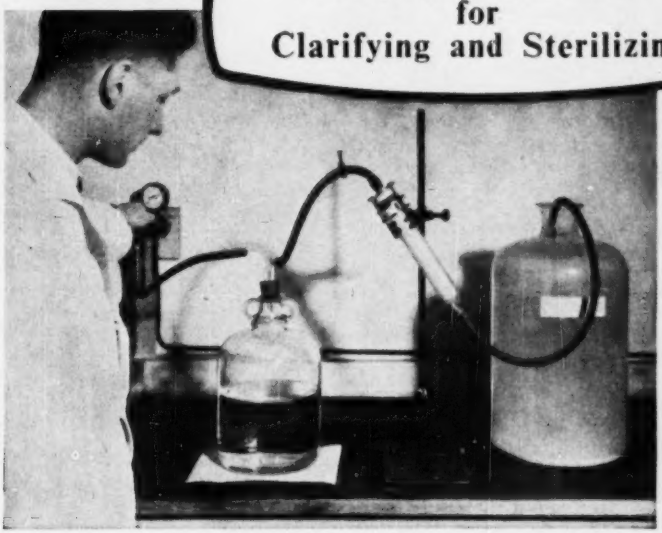

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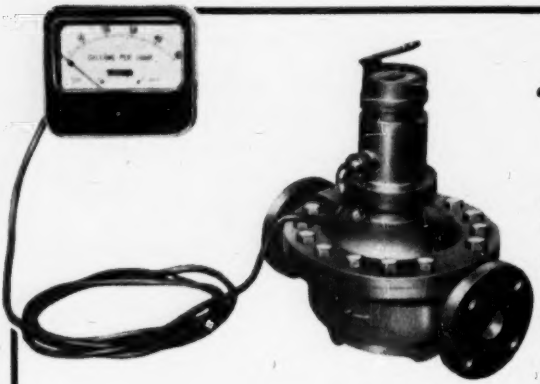

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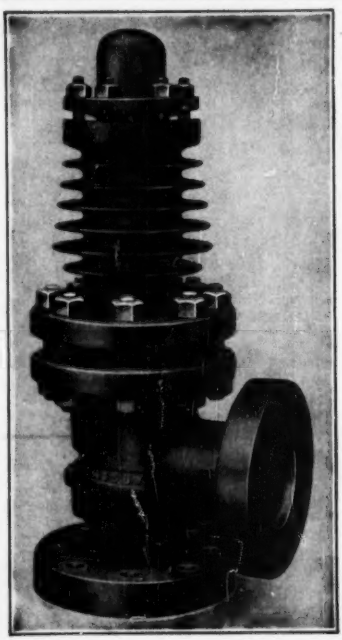

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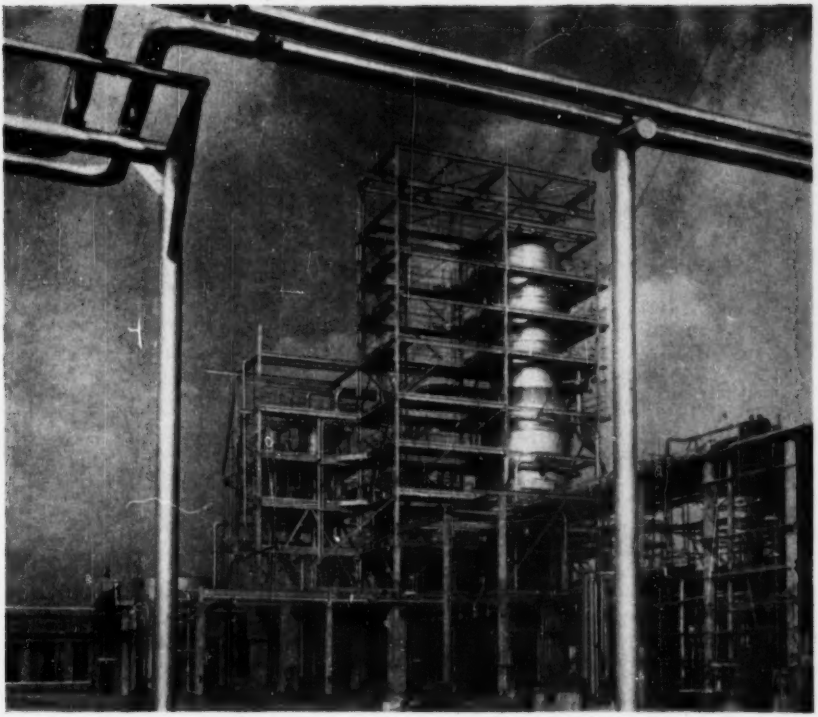

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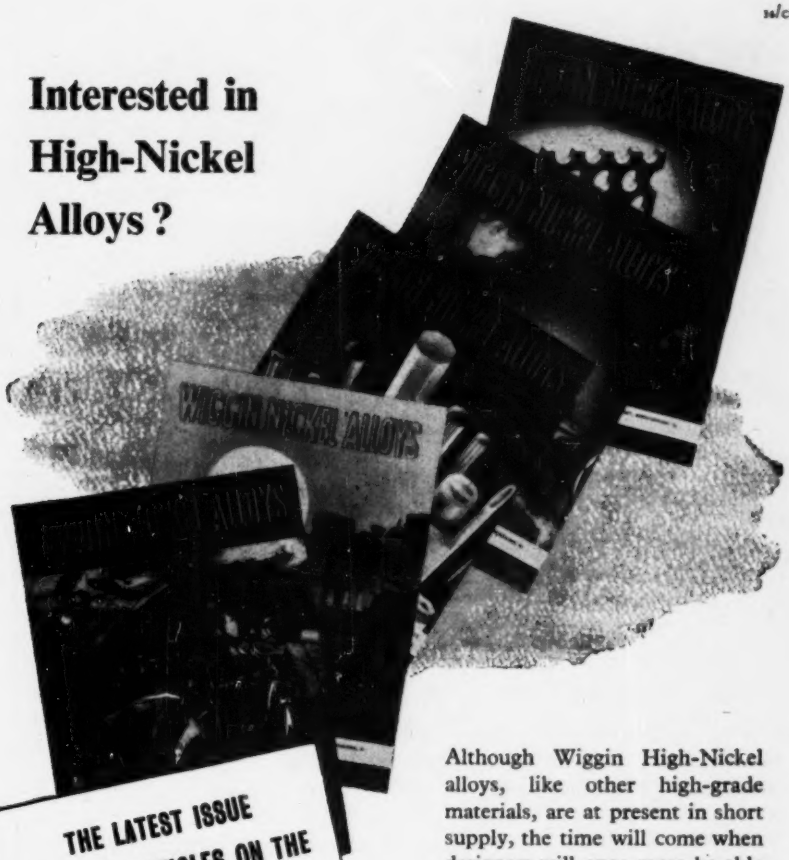

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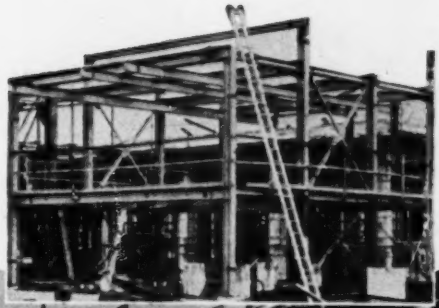

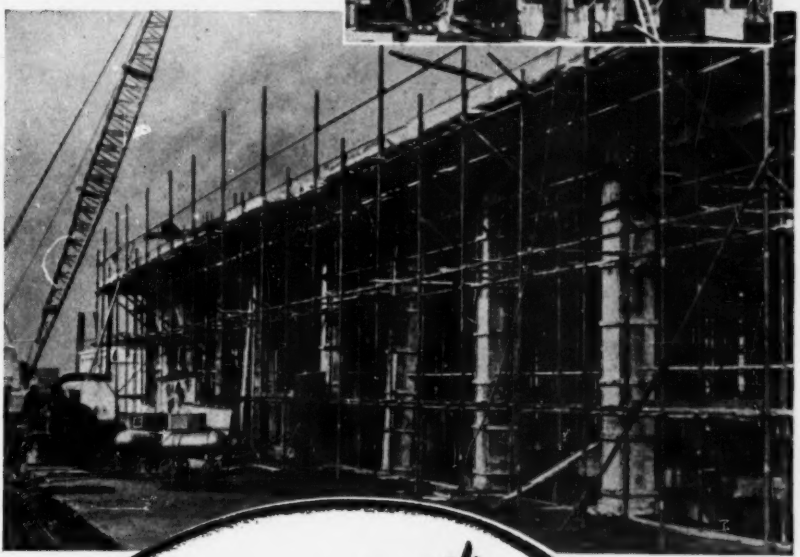

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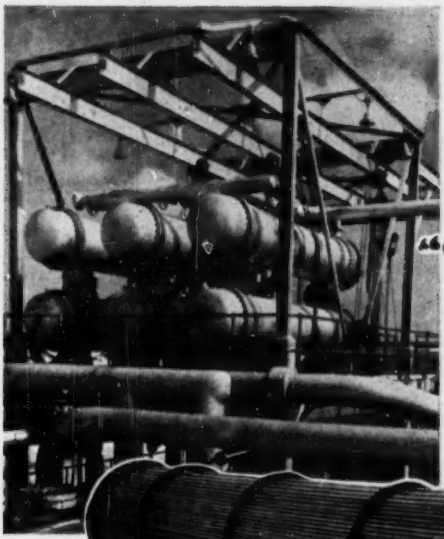

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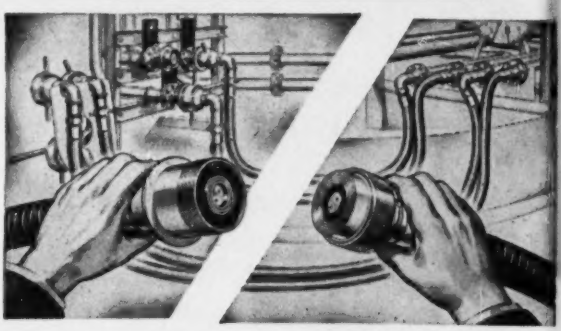

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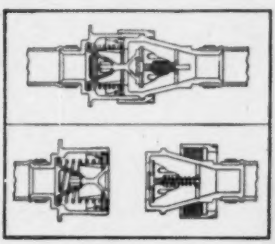

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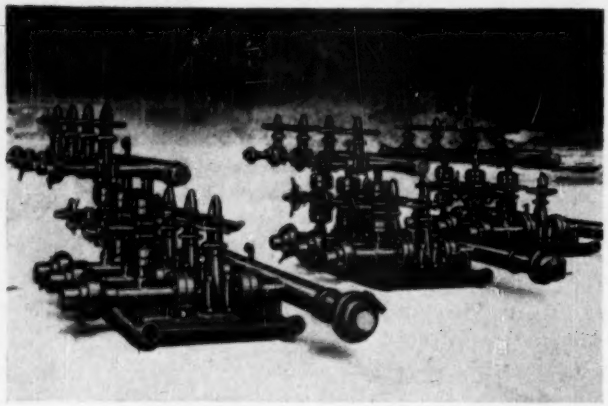

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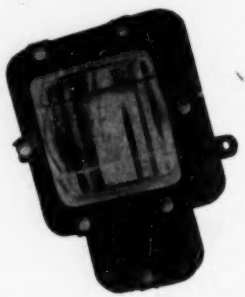

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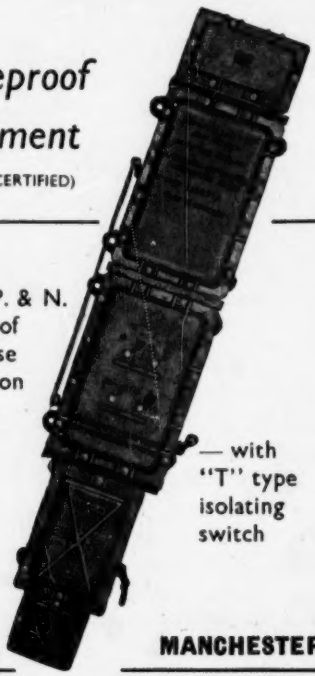

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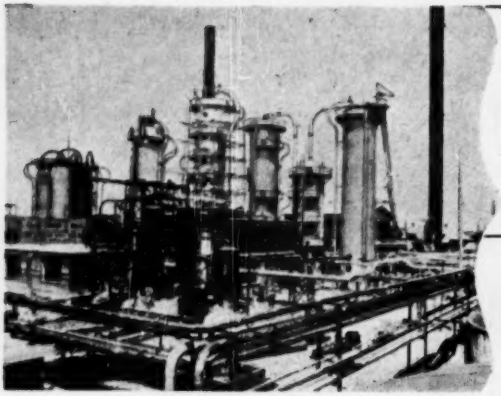

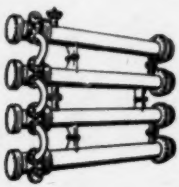

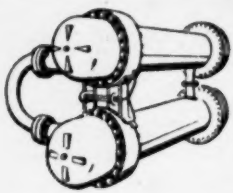

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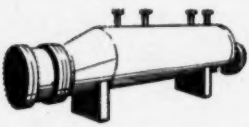

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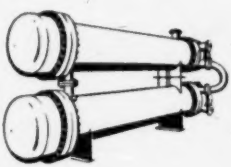

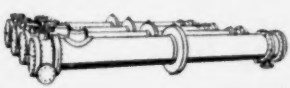

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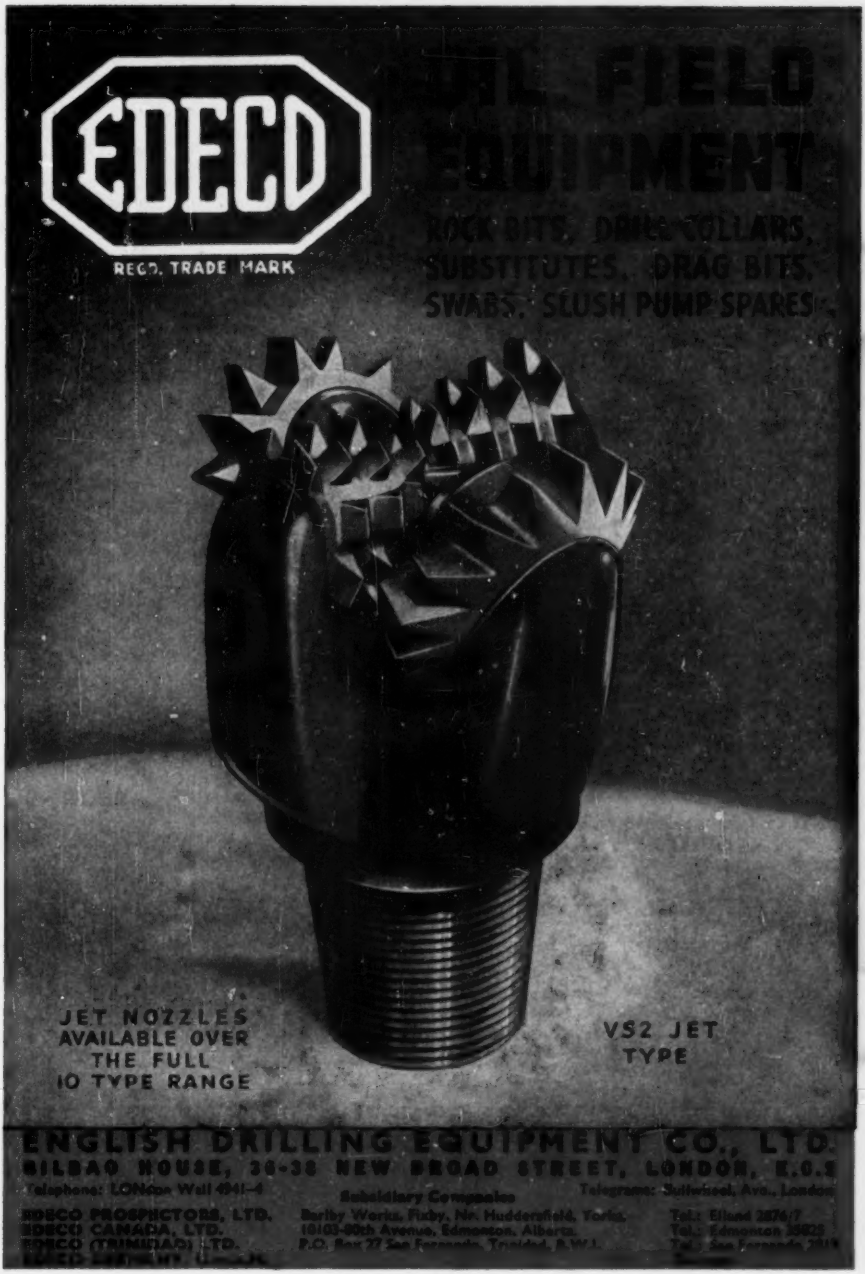
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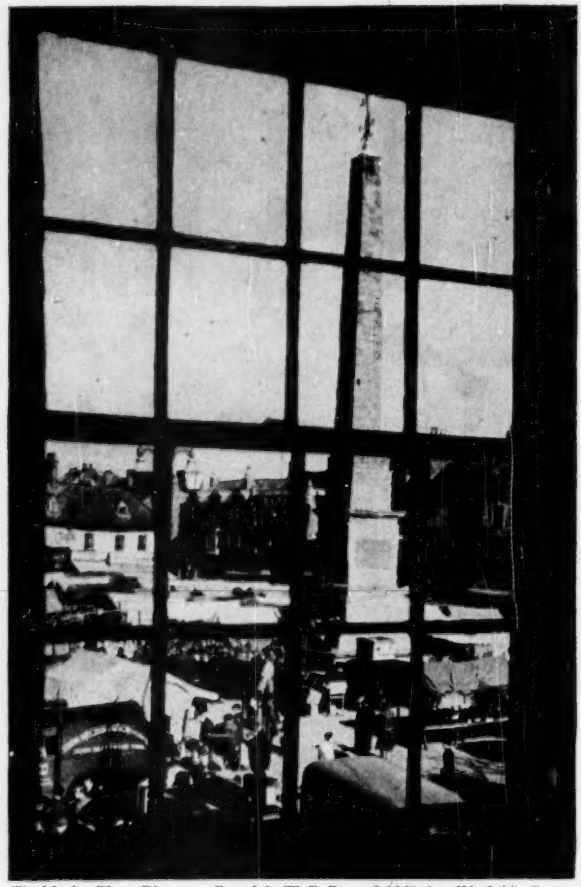

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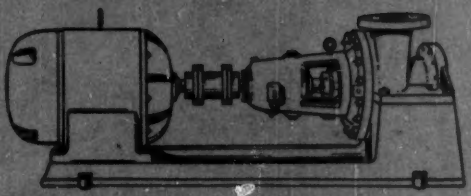

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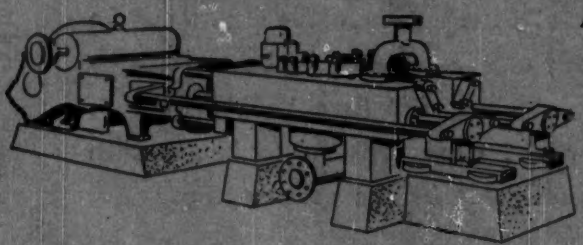

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